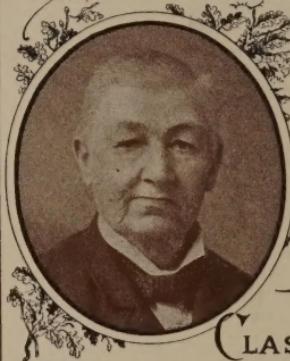


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ZINC OXIDE

*History, Manufacture and Properties
as a Pigment*

BY

DALTON B. FALOON, B. S.

—
Illustrated
—

FIRST EDITION



D. VAN NOSTRAND COMPANY

EIGHT WARREN STREET

1925

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*Jan 1926
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SINCERELY DEDICATED
TO MY DEAR FRIEND
Dr. Edgar Fahs Smith

75553

PREFACE

A FEW years ago, while in the plants of The New Jersey Zinc Company, I searched exhaustively for information on the subject of zinc oxide. I had the opportunity of reading all the books on zinc and zinc oxide that were in the New York Public Library—however, they were few in number and meagre in enlightenment. Search was made through libraries in Philadelphia, Chicago, and other large cities. It became plainly evident that even though a chapter or a few pages of a book did describe the pigment, a complete comprehensive work dealing with zinc oxide had never been written.

My voluminous notes, both from plant experiences and personal researches, were collected, classified and arranged for the writing of a treatise on zinc oxide. I have endeavored to tell logically the complete story of this widely used pigment, making my treatise technological rather than technical. Complex and highly technical details have been omitted and the data and facts have been aranged in an understandable manner. Illustrations have been freely used to save the readers' time and to more clearly depict the plant, machinery or procedure being described.

The book has been designed and arranged to help the man who *uses* zinc oxide in the paint, rubber, oil-cloth, ceramics, printing ink, linoleum, etc., industries, and I believe will prove a help in many ways. This

being the first book ever written solely on the subject of zinc oxide I am fully cognizant of the fact that there are undoubtedly many matters in it which may require alteration for future editions, and probably many sections that shall necessitate enlargement.

The following works, among others, have been consulted and my general indebtedness to these authors must here be thankfully recorded: Hofman, "Metallurgy of Zinc and Cadmium"; Smith, "The Zinc Industry"; Lones, "Zinc and Its Alloys." I acknowledge gratefully the assistance and suggestions given by my friends in the manufacturing departments of the various industries.

It is hoped that the data contained in this treatise will be of value to all who are interested in this pigment.

DALTON B. FALOON

Cleveland, Ohio

October, 1925

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INTRODUCTION

The zinc industry in the United States has been surrounded by an atmosphere of mystery. Information and facts relative to common principles and practices have been closely guarded, the manufacturing plants have been as safely barricaded with fences and walls as though they were rare golden treasures, and the few people who have known the industry in all its branches, were prompted to keep their information from channels of circulation.

During the past few years the industry has grown by leaps and bounds and the use of zinc, and especially zinc oxide, has become very common. The advent of the motor car and the dependence of the rubber industry upon zinc oxide developed a gigantic market for this pigment. The educational work of the progressive American paint manufacturers has brought the painters and the average home owners to recognize the superiority of a paint composed of both white lead and zinc oxide. Later developments are indicating that new pigments designed to replace white lead may prove worthy of consideration, but they can not be used in a practical way without being combined with zinc oxide. Thus zinc oxide has attained a position of both permanence and prominence.

The users of this pigment have not been given the opportunity of becoming familiar with the processes of

manufacture, and, therefore, do not fully understand the nature of the pigment. They have recognized certain qualities and properties and by the continuous use of zinc oxide they have become familiar with its applications, but there has never been any attempt to give them the interesting story of zinc oxide before it reaches their plants.

Each user of zinc oxide has had his particular method of testing the material to be certain that he can use it with safety and to be certain that he is maintaining a commercial standard in the quality of his products. This lack of standard specifications and standard methods of testing both physically and chemically naturally resulted in general confusion and misunderstanding among the various users and producers, and has given a very unsatisfactory basis for a gigantic industry.

The present work is a step toward arranging general information concerning this industry, in such a manner that all who are interested may know the complete story of zinc oxide. Care has been taken to point out where and why, in the manufacture of zinc oxide, the particular qualities are determined.

Specifications for each grade of zinc oxide have been suggested. Standard methods of physical and chemical tests are arranged. These methods have proven to be the best for standard procedure and may be adopted with confidence.

This book was not written as the final work on zinc oxide or the zinc oxide industry. It is hoped that its publication may pave the way to having more information made available to all who may seek knowledge concerning zinc oxide.

ZINC OXIDE

CHAPTER 1

THE HISTORY OF ZINC

The history of zinc begins at very early times. Zinc has been found in such places as to indicate that it was used by the early Greeks, Romans and Arabians. There is conclusive evidence that in very early days zinc-bearing ores were used and condensed furnace fumes were used to produce zinc alloys and to make compounds for medical purposes.

“False silver,” (*pseudargyros*, in Greek) was the first description given of zinc, by Strabos in the passage describing Andriera in Mysia. Thus it is asserted that the ancients were acquainted with zinc in its metallic state. This fact seems to be substantiated by recorded recoveries and discoveries of antiquities, said to be metallic zinc. It is possible that the passage of Strabos is too vague to give guidance for historical study, but it is interesting to note that near Balia, not far from the site of Andriera, there occur zinc deposits of blende, zinc sulphide and iron pyrites and galena.

The oldest known piece of zinc extant is in the form of an idol found in the prehistoric Dacian settlement at

Dordsch, Transylvania. The analysis of the idol showed Zinc, 87.52 per cent; Lead, 11.41 per cent; Iron, 1.07 per cent. In 1772 a small metallic bar was found at Campagne in the ruins of the old Roman town. M. Grignon, who examined the bar, concluded that it was zinc and stated that the form of the crystallization of the metal indicated that the bar had been "worked" to some extent. He also stated that in the vicinity of the Roman town there was an iron mine containing a large amount of Zinc.

The Ancients, it has been held, were not acquainted with zinc. However, in the ruins of Pompeii, destroyed A.D. 79, there was found the front of a fountain, the upper section of which was covered with zinc. A really valuable record is made by M. Salzmann relative to the discovery of two bracelets in the ruins of Camirus, destroyed 500 B.C., in the Isle of Rhodes. He says: "The silver trinkets are sometimes solid, sometimes plated; certain bracelets for example, were made of a hollow silver ring filled, not with tin, as I believed at first, but really with zinc."

The Greeks were not acquainted with the production of brass, as were the Romans, as early as 200 B.C. Brass was prepared about the time of Augustus, 20 B.C. to A.D. 14 by mixing zinc bearing wall accretions, zinc oxide ore and small pieces of copper—these materials being slowly fused in a crucible. In this operation the zinc oxide was reduced to metallic zinc and then the temperature raised to assure fusion of the metals. Brass made in this manner was used for coins, ornaments, etc.

Thus it was highly probable that zinc as a metal was known to the ancients, although its production

and use was only occasionally known and the nature of the metal was very little understood.

The medieval history of zinc is bound up with the history of alchemy of that period. However, inferences drawn from the works of alchemists of that period are very unsatisfactory. The production of brass—the yellow, gold colored alloy of zinc and copper—was well known to the alchemists and the knowledge gave rise to the belief that by the use of zinc, and zinc-bearing materials, it might be possible to transmute copper into gold. This belief induced them to secrete the results of their researches and if they wrote at all on the subject of zinc they often tried to confuse rather than enlighten their readers.

In Europe the word *zinck* first appears in the works said to have been written in the fifteenth century by Basilius Valentinius. In the third book of the Strasburg edition (1645) of the "Last Testament of Basil Valentine" occurs mention of ores, metals, minerals, zincks, etc. But there is nothing to show that he actually meant metallic zinc. Actually the first writer to give the name *zinck* to the metallic form of the metal was Paracelsus (1490–1541). Many passages in his works (i.e., "Hermetic and Alchemic Writings") show that he was acquainted with the metallic nature of zinc, that he knew it was fusible but not malleable, and his writings indicate that he was rather well informed regarding all the physical qualities of zinc.

Zinc continued to be a rare metal in Europe for many years after the time of Paracelsus. In India, however, metallic zinc was extracted long before the time of Paracelsus, and the Hindus were acquainted with a method of extracting the metal by a process of dis-

tillation downwards into a vessel containing water. Knowledge of the production of zinc was also known earlier in Asia than in Europe. Kazwiui, called the Pliny of the Orient, who died A.D. 630 stated that the Chinese knew how to render the metal malleable and that they used it for small coins and mirrors.

During the seventeenth and eighteenth centuries large quantities of slab zinc, or commercially called spelter, were imported from the East. Various names were given to this metal, such as Indian tin, calaaem, tutaneg and spiauter. The account books of the various companies trading in India, Sumatra and China give a great deal of information about this trade but the actual dates of its inception are not known. It was before the year 1611 for in the beginning of the seventeenth century, namely 1620, a Portuguese ship carrying spelter from the East Indies was seized by the Dutch. This metal was sold in Paris and other places under the name of *speautre* or *spialter*. The name was latinized to *speltrum*, from which comes *spelter*, the commercial designation for slab zinc. In a letter dated Sept. 14, 1611, Libavius clearly shows that at that time the calaaem (spelter) of Malabar was known to him and he says that it seemed to be the same as zinc.

About the year 1740 Dr. Isaac Lawson brought the knowledge of smelting zinc from China and under his guidance John and William Champion set up a smelting works at a brass plant near Bristol, in England. The process was for producing spelter by distillation, *per descensum*. The process was kept secret until toward the end of the century when Johann Richberg visited England, learned the art of making zinc, and returned to Upper Silesia. In 1799 he started a plant

in operation near Myslowitz. Now Upper Silesia is one of the most important zinc centers of Germany. In 1806 zinc smelting was begun at Liège, Belgium, and there the foundation was laid for the Vieille Montagne Co. success in 1837.

In the United States the first zinc was produced in 1835 by John Hitz in the Arsenal in Washington. It is said that the United States Government, in the desire to establish definite standards of weights and measures, imported workmen from Belgium, built a small spelter furnace at Washington to make the zinc necessary to form the brass needed for standard units of weight and measure. The ore used was zincite from the northern portion of New Jersey. The pit from which the ore was taken was known for years as the "Weights and Measures Opening" and remained intact until about the year 1900 when continued mining operations caused its disappearance.

The first American attempt at commercial production of zinc was begun at Newark, N. J., in 1850, a Belgium zinc furnace being used. The ore used was Franklinit (zinc-iron-manganese ore) and the attempt was very unsuccessful as the iron content of the ore corroded the retorts. In 1856 another zinc plant was built at Friedensville, Pennsylvania, but similar troubles presented themselves due to the ore. In 1860 J. Wharton erected a zinc plant at South Bethlehem, Pennsylvania, and it proved to be a success. Thus it was in the Lehigh Valley of Pennsylvania, which now holds the world's largest zinc plant at Palmerton, that the zinc industry emerged from experimental to the commercial plane. From this point new zinc plants seemed to take life—and in 1862 a successful plant was

operating at La Salle, Illinois. Then followed several zinc works at St. Louis, Missouri and at Mineral Point, Wisconsin; later at Weir City and Pittsburgh, Kansas. At the present time the leading smelters are found in Oklahoma, Illinois, Pennsylvania, Kansas, Arkansas and West Virginia.

The commercial production of zinc oxide as a pigment was started in France in the latter part of the eighteenth century by LeClair and Sorel. LeClair was a paint grinder and master painter. He was using white lead as the main pigment and he noticed that there was a great deal of sickness among his workmen due to lead poisoning. In order to overcome this he made some elaborate and exhaustive tests with zinc oxide as his pigment. He found that this pigment not only overcame the illness of his employees but gave him a whiter and more desirable paint. Investigations of LeClair's claims by the French Government led it to specify Zinc Oxide for all government work. Later white lead was prohibited above certain percentages and zinc oxide specified in its place.

The zinc oxide used by LeClair was made by burning spelter (slab zinc) with an excess amount of air. The plant he erected and used was still in operation up to the breaking out of the World War. The process used is known as the French Process, or the indirect process, since in its operation spelter must be produced from the ore and then "burned" in a special furnace to produce the zinc oxide.

The manufacture of zinc oxide in America is closely connected with some of the early endeavors to solve the problems of successfully using the complex refrac-

tory and little known ores from the unique deposit in Northern New Jersey.

The deposit of mineral ore in Northern New Jersey was discovered the latter part of the eighteenth century by a party of Swedish miners who were traveling overland from Baltimore to New York. The earliest record that we have of this deposit of ore, however, is 1824 when some of the minerals occurring there were described by Messrs. Van Uxem and Keating. The first mining that was done from this deposit was at the time when the United States Government made its standard weights and measures, as mentioned previously. Real mining operations did not begin, however, until about 1850.

The deposit is of complex ore containing mixtures of franklinite, zincite, and willemite—a hopeless combination for producing either spelter or iron. The fact that zinc oxide could be made from this ore is said to have been discovered about 1854 by a workman by the name of Burrows, who was employed by the Passaic Chemical Company at Newark, N. J. There was a zinc plant adjoining the plant of the Passaic Chemical Co. and Burrows, while on night duty, discovered a badly leaking fire flue in one of the furnaces he was attending. He mended this hole by throwing an old piece of fire grate over the exposure, and shoveled over it some of the zinc company's mixture of ore and coal, which he found handy. He was surprised a short time later to see a cloud of zinc oxide arising from the burning patch. He later related his experience to Messrs. Wetherill and Jones, who were connected with the zinc company. His discovery led to developments

along this line and to the many patents taken out by Wetherill between 1855 and 1868. Mr. Jones perfected a method of collecting the zinc oxide by means of fabric bags. The processes developed by these two men have been used ever since for the manufacture of American, or direct, process zinc oxide.

CHAPTER 2

ZINC ORES—LOCATION AND SUPPLY

Minerals that bear zinc are very widely distributed geographically—so widely, indeed, that they are found in almost all the metal bearing districts of the world.

The minerals which form the basis of ores are comparatively few in number. The ores of zinc are found more particularly associated with the ores of lead, copper and silver, often forming large and extensive deposits of great complexity, which present serious problems to the metallurgist, for the practical solution of which a large number of processes have been devised. The ores of zinc may be classified as sulphides (blende, wurtzite), as oxides (zincite, franklinite), as silicates (willemite, calamine), as carbonates (smithsonite, hydrozincite), and as sulphates (goslarite). In addition to these classifications may be mentioned cadmia, the throat accretions of blast furnaces treating zinc-bearing iron ores.

While zinc occurs as a constituent of a very large number of the ores of other metals, minerals containing the metal in sufficiently large quantity and occurring in sufficient abundance to be really ores of zinc, are very few. The metallurgy of zinc requires the employment of ores analyzing relatively high in zinc content. It is, therefore, frequently necessary to submit zinc-

bearing ores to some preliminary process for the concentration and separation of the zinc mineral from other minerals and gangue, or worthless material, before a product sufficiently high in zinc content can be obtained ready for use by a zinc smelter. The ore mined is frequently very low in zinc content, often averaging not more than 3 per cent, but this percentage is increased by concentration to 40 per cent zinc or often more.

The principal zinc ores are as follows:

Blende and *Wurtzite*, average about 67 per cent zinc and 33 per cent sulphur. The mineral ore blende—called in manufacturing classifications, sphalerite, Jack, rosin Jack, blackjack—forms isometric crystals and the mineral ore Wurtzite forms hexagonal crystals. Blende, the sulphide formation of the mineral, is rather common; wurtzite is rather rare, although it has been found to some extent in some Missouri and foreign ores. The most common deposits of blende contain little iron and has a reddish brown color, the more iron contained in blende the darker the color. If the blende contains as much as 10 per cent or more of iron, as FeS, the color is dark brown to black and this high iron bearing blende often goes under the name of black blende or marmatite. In addition to iron, sulphides of copper, cadmium, arsenic, mercury, silver, and gold minerals and oxides of tin are also found with blende. The gangue, or waste material, associated with blende is mostly of limestone formation, but sometimes it is silicious. Blende ores, being the most common, are usually low grade. The following chart shows the average analyses:

	Joplin, Mo.	Sheet Ground, Mo.	Alber- marle, Va.	Okla., Kans.	Park City, Utah
Zinc.....	58.26	59.00	62.11	58.60	46.00
Cadmium.....	.30	.058			
Lead.....	.70	.293	1.04	1.86
Copper.....	.049	.054	1.54
Iron.....	2.23	1.9	3.44	1.6	7.4
Mn.....	.010	None	2.0
CaCo ₃	1.880	Trace			
CaO.....05	.90	4.90
MgCo ₃850	None			
Mgo.....	None		Trace
BaSO ₄820	Trace			
SiO ₂	3.950	6.971	.26	5.20	4.10
NiCo.....	None	Trace			
S.....	30.420	31.301	34.45	31.28	30.40
As.....52
Sd.....	
Al ₂ O ₃	5.20
Ag.....	

These ores, having a relatively high sulphur content, must first be treated to free the mineral of sulphur. The sulphur is removed by roasting the ore either in a kiln consisting of longitudinal shelves through which the ore is raked from shelf to shelf, or a kiln consisting of circular shelves through which the roasting ore is raked downward, shelf to shelf. The sulphur is freed at the top of the kiln and goes off as SO₂, is conducted through a series of coke and felt cleaners and used to manufacture sulphuric acid. The analyses of several blende ores before and after roasting are as follows:

(Raw Ore)	SiO ₂	Zn	Fe	Pb	Sul.	CaO	Mn	Mg	Cd
H. G. Separator.....	1.5	57.3	2.3	1.5	31.3	2.3	.03	.80	.008
Missouri.....	6.4	55.9	3.7	0.9	29.8	0.8	.14	.17	.032
Wis. separator.....	2.3	58.3	2.2	0.7	31.5	2.5	.21	.74	.009
Mexican concent.....	1.7	59.0	2.2	0.6	31.5	2.0	.08	.65	.083
Wellington.....	3.4	47.9	10.5	1.6	30.2	0.9	.63	.21	.023
Cañon City.....	4.8	44.6	8.6	7.0	29.8	0.7	.56	.50	.023
Broken Hill.....	2.0	46.8	9.7	6.4	30.2	0.3	1.40	.24	.011
(Roasted)									
H. G. separator.....	.9	66.6	3.0	1.0	2.2	2.8	.05	1.41	.015
Missouri.....	5.4	66.8	2.4	1.2	3.1	1.0	.04	.28	.036
Wis. separator.....	1.6	63.2	2.9	0.9	4.9	3.8	.14	.93	.009
Mexican concent.....	2.9	41.7	10.8	3.6	7.0	1.6	..	.30	
Wellington.....	4.0	55.0	11.4	1.6	2.4	1.2	.70	.24	.021
Cañon City.....	5.0	50.9	11.5	3.0	1.9	1.3	.42	.35	.023
Broken Hill.....	4.2	52.1	10.1	6.5	2.0	0.8	.68	.13	.013

Zincite, Franklinite and Willemite are three zinc ores found intermixed in one deposit at Franklin, New Jersey. Zincite is primarily a natural formation of zinc oxide. It is the richest zinc ore known, containing about 80 per cent zinc. It is of deep red to orange yellow hexagonal mineral, the color being due to about 5 per cent manganese. Franklinite is a black ore composed of the combined oxides of iron, manganese and zinc. It forms rounded octahedral crystals, and is sufficiently magnetic to permit magnetic separation, from zincite and willemite. The Franklinite concentrate contains about 39 per cent to 46 per cent of iron; 10 per cent to 19 per cent of manganese and about 22 per cent of zinc, with about 4 per cent of silica. Willemite is a yellowish green mineral composed of zinc, silica and oxygen in the form of orthosilicate of

zinc. It also contains manganese to the extent of 1 per cent to 3 per cent.

These three minerals are found in Northern New Jersey in cambian limestone. Two mines, one at Franklin and one at Sterling Hill are worked. The ore is milled, and then subjected to magnetic separation to segregate the franklinite and is later subjected to wet concentration by both jigs and tables to recover zincite and willemite. The franklinite is used to produce zinc oxide, and the clinker of the process is later used in blast furnaces to produce a manganese-pig iron, known as spiegeleisen. The zincite and the willemite are used to produce spelter.

Calamine, Smithsonite, Hydrozincite.—Calamine is a white mineral ore having a bluish green shade. It forms ortho-rhombic crystals and has a glassy luster. It will analyze about 57 per cent zinc. Smithsonite is also a white mineral, but may appear in greenish or grayish casts. It forms curved and imperfect rhombohedral crystals. It will contain about 52 per cent zinc. Hydrozincite is white to yellowish in color and forms earthy incrustations. It usually contains about 47 per cent zinc. These three minerals usually occur together in oxide formations and are known as the carbonate ores. The first, calamine, is relatively common; the second, smithsonite, is rather rare in the United States but common in Europe; the third, hydrozincite, is also well known.

Goslarite and Cadmia.—Goslarite is found in the form of white vitreous ortho-rhombic crystals. It contains about 23 per cent zinc. It is a secondary mineral resulting from the alteration of blende, but is rather important as an ore. Cadmia is contaminated zinc

oxide that forms as throat accretions in iron blast furnaces in the treatment of zinc-bearing iron ores. It usually contains about 85 per cent to 95 per cent zinc in the form of zinc oxide.

Zinc Ores of the World

Zinc minerals are widely distributed geographically as is shown by the accompanying map, on which the principal zinc ore-producing districts are indicated.

British Empire Zinc Ore Deposits

In the United Kingdom the best known zinc deposits are those of Flintshire, Cardiganshire, Durham, Cumberland, Derbyshire, Isle of Man, Dumfriesshire and Lanarkshire. The mineral mines are worked for both lead and zinc, and the zinc ore produced is mostly blende.

In Canada there are many deposits of zinc-bearing ores but the output is not large. Most of the zinc ores coming from Canada are from British Columbia, mainly from the Kootenay district. The ores are very complex and hard to use.

Australia has well known and relatively important deposits of zinc ore at Broken Hill, New South Wales. These are the most important deposits of the British Empire. The ore is a complex association of galena and blende. The ore produced mainly goes to Continental Europe. In pre-war times these deposits produced enough ore to satisfy one-fifth of the world's trade needs of zinc.

Tasmania has deposits of lead sulphide ores in Mt. Read district on the west coast of the Island. The

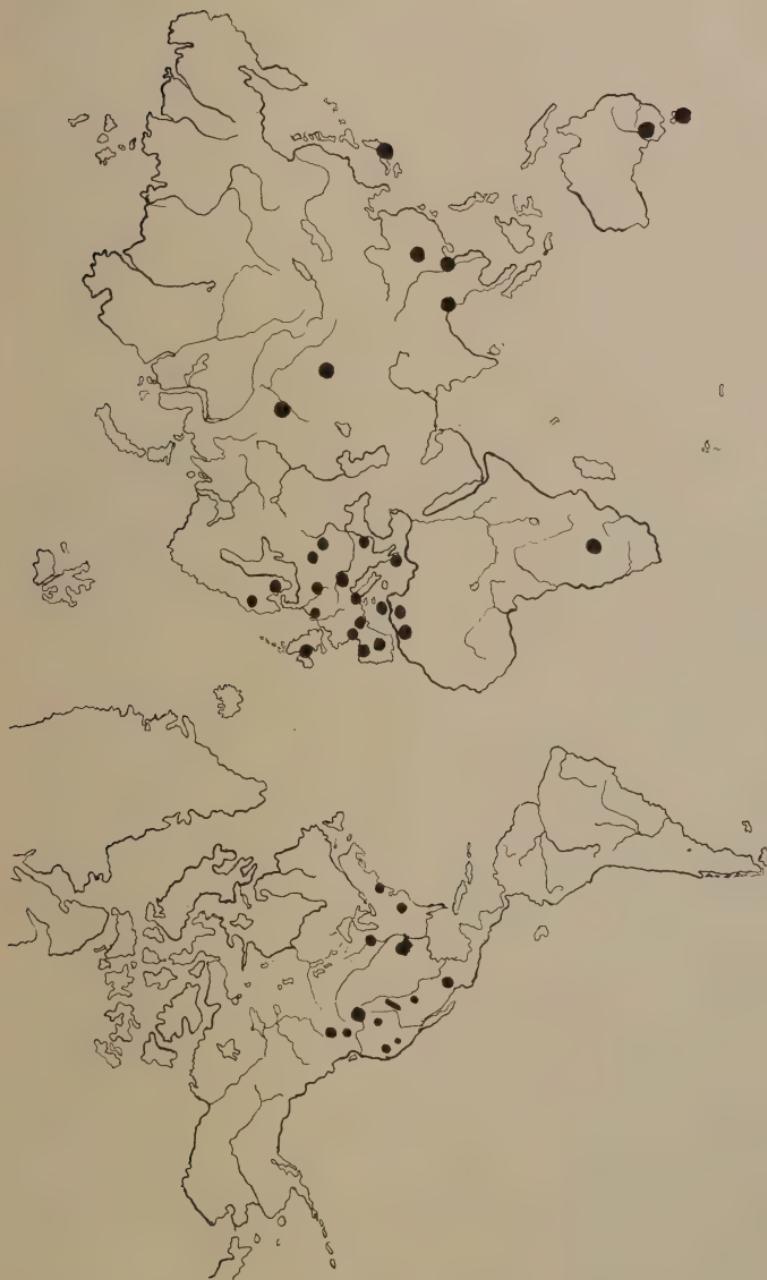


FIG. 1.—The world's zinc ore deposits.

zinc-lead-sulphide deposits occur over a length of seven miles and contain 24 per cent to 42 per cent zinc.

India has large and prospective deposits of silver-lead-zinc ores of the sulphide class in Upper Burma. These deposits are situated at Baldwin in the North Shan States, some sixty miles from the Chinese Province of Yunnan. They also contain an appreciable amount of silver.

South Africa has a lead and zinc deposit at the Rhodesian Broken Hill District. A discovery of zinc-bearing ore in Northwestern Rhodesia is said to look promising.

European Sources of Zinc Ores

Austria has important zinc ore deposits situated in Southern Carinthia, Styria and Tyrol.

Belgium has been a small producer of zinc ore since 1856, but it was formerly a very important producer. The remaining important zinc mines are at Blyberg, and near Verrières and Liège; also the Vielle Montagne mines at Monset.

Bulgaria is a small zinc ore producer. The main mine is the Sedmolchislenzia Mine about six miles southwest from Vratza.

France first produced zinc ore about 1870, but the output has fallen off very much. The largest mines are now at Malines. In the Pyrenees zinc ore occurs many places. Sientien Mines in Ariège produce carbonate ores of zinc and lead. Deposits also occur in Hantes and Basses Pyrenees. Also at Var, where silicate, zinc blende and zinc carbonate occur. The French colonies of Algeria and Tunis also produce zinc. In

Algeria, in the Department of Constantine, zinc-bearing ores are found, and in several places along the Atlas Mountains zinc ores are also mined.

Germany is the chief producer of zinc ore in Europe. The most important deposits are situated in Upper Silesia, while ore in smaller quantities is obtained in Rhineland, Westphalia, Nassau, the Harz, etc. The well-known deposits of Upper Silesia occur in the southeastern corner of the Province, on the border of Austria and Russia. They are principally calamine and blende ores.

Greece has zinc ores in the Laurium district. They are zinc carbonate and blende ores.

Italy has deposits of zinc carbonate ore at Sardinia. Less important deposits occur in Lombardy, Piedmont and Tuscany.

Norway has small amounts of zinc ores at Hadeland, Modum and Ranen. There is also a large deposit of lean, low zinc content ore near Christiana.

Russia has zinc ores in Russian Poland district near Silesia—producing calamine. This is probably an extension of the Silesian deposit of ore. Spain has ore deposits in the provinces of Murcia and Santander. They are not relatively important.

In Sweden the only important mines are those of Ammeberg, in the Nerike Province.

Asiatic Sources of Zinc Ores

Little accurate information appears to be available regarding the nature and extent of many of the zinc deposits of Asia.

China has the most important deposits. These

deposits, which are worked, are in the Prefecture of Changlin, in the Hunan Province.

Japan.—The most important deposit is at the Kamioka Mines, Province of Hilda. Zinc mines of lesser importance occur in the Provinces of Tsushima, Etchu, Echizen, Bizou and Mimasaka.

In Siberia important deposits are worked at the Ridder Mine in the Altai Mountains.

Zinc Ores in the United States

Zinc ores are to be found in many regions of the United States. The deposits, however, may be classified as belonging to defined areas of Eastern, Central and Western Zones.

The Eastern Zinc District

The Franklin, New Jersey, deposit is the principal zinc ore area of the Eastern District. This deposit is mined at Sterling Hill and at Franklin, N. J. About eighty different minerals have been found in the Franklin Mine, the principal ores of which are: Franklinit, willemite and zincite. With these minerals are to be found gangue or useless materials of calcite, rhodenite, garnet, pyroxene and hornblende. This ore body at Franklin, N. J., is considered to be the unique mineral deposit in the world. Over seventy-five specimens of different minerals have been found in this deposit, which is a vertically inclined, hook shaped long deposit of solid ore. The ore body is so situated that efficient mining methods can be practiced with facility. By having systematized stopes and pillars all of the ore will be mined in the course of years, and

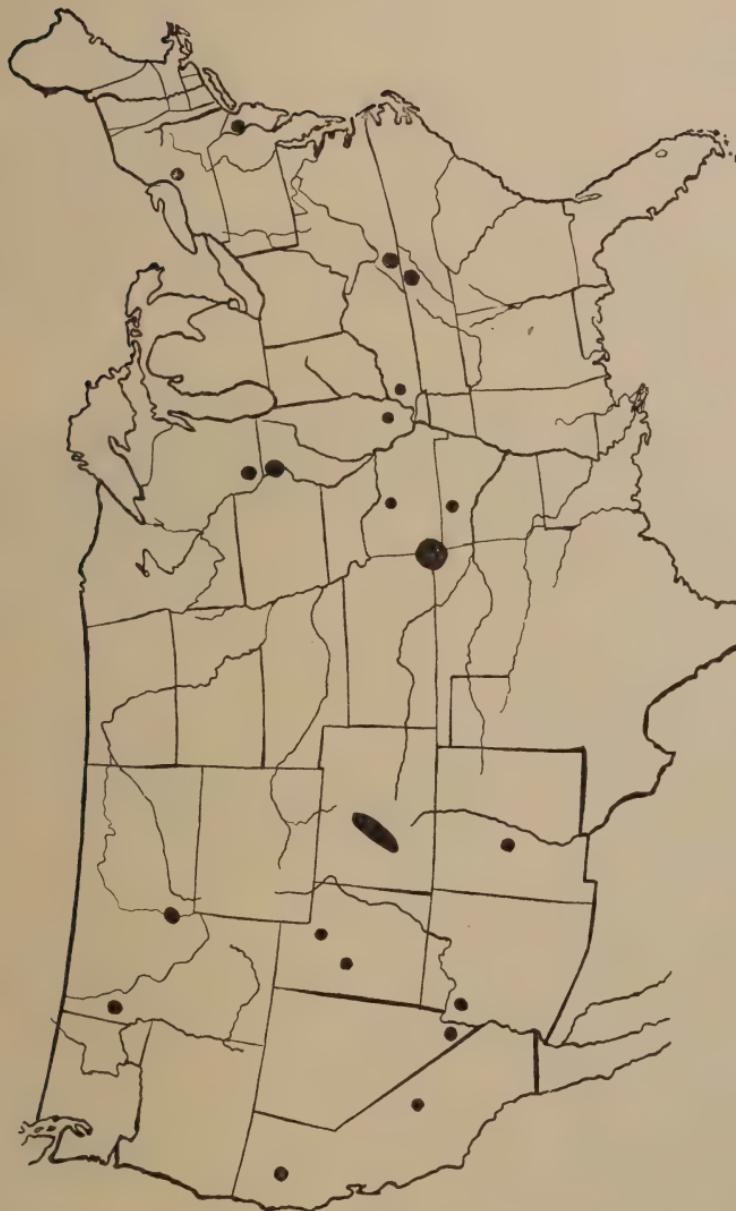


FIG. 2.—Zinc ore deposits of the United States.

the resources of this valuable deposit will have been used with conservatism.

The only other zinc-producing region in the Eastern District is the Virginia-Tennessee area. The zinc ore deposits occur in a limestone formation extending from Southwestern Virginia into Eastern Tennessee. In Virginia the Bertha and Austinville mines have been the chief producers, the ores mined being principally weathered ores, with calamine, smithsonite and cerrusite as the metal-bearing minerals. The ores are concentrated so as to analyze about 40 per cent zinc. The gangue minerals are calcite, barite and dolomite.

Tennessee zinc ores are more abundant. Both oxide and sulphide ores are obtained, some being lead-bearing ores and others being practically lead free. The better known of the Tennessee ores is from the Mascot mines and contain about 10 per cent blonde, no iron and no lead. By wet concentration these ores can be brought up to about 60 per cent zinc. The lead free Tennessee zinc ores are generally high in cadmium content but are refined by a method which employs bone black, and the ores thus produced are used for the manufacture of lead free zinc oxide.

New York State also produces zinc ore but the quantity is relatively unimportant.

The Central Zinc District

The Central Zinc Producing District covers a very large area embracing the Upper Mississippi Valley, the Ozark region, and the smaller areas in Northern Arkansas, Kentucky and Illinois.

In the Upper Mississippi Valley region zinc ore is pro-

duced in Southwestern Wisconsin, Eastern Iowa and Northwestern Illinois. Wisconsin is the principal producer of the Upper Mississippi Valley. The ores are found in the galena, dolomite and the upper part of the Plattesville limestone, both of the Ordovician Age; blonde, in some places, is inter-mixed with marcasite and must go through a roasting process for magnetic separation in order to remove this mineral from it. The raw ores will analyze about 12 per cent blonde, and after concentration will contain about 60 per cent zinc. Blonde is the principal ore produced by this section. The ores of Eastern Iowa and Northwestern Illinois are very similar to those of Wisconsin.

The Ozark zinc-producing region lies mainly in Missouri, with its center around Joplin, but also includes portions of Arkansas and Oklahoma. The ores are found in Southwestern and Central Missouri, Northwestern Arkansas and Northeastern Oklahoma. The ore is found in limestone and chert formations, combined with dolomite, calcite and blonde, also containing blonde, galena and marcasite. The ore bodies form *runs* and sheet ground. The runs are irregular tabular ore bodies, 10 to 50 feet wide and rather shallow. The sheet ground has a similar formation but it is greatly broadened out and is from 15 to 16 feet thick. In this area an ore body carrying 6 per cent zinc is considered valuable. After concentration ore will analyze about 55 per cent to 60 per cent zinc. The zinc ores found in Central Missouri, Arkansas, Kentucky and Southern Illinois are not as important as the Missouri-Kansas-Oklahoma district that centers around Joplin.

The Western Zinc District

The states of Colorado, New Mexico, Montana, Idaho, Utah, Arizona, Nevada and California are the leading zinc producers of this region.

The Colorado zinc ore deposits occur mainly in a belt extending from the Rico district in San Juan region northeasterly to the central district of Boulder County. In the Leadville region zinc carbonate and silicate are found below carbonate lead formations in the carboniferous limestone. This ore will analyze about 30 per cent to 35 per cent zinc and 3 per cent to 6 per cent lead.

The New Mexico zinc ores come mainly from the north and western part of the state. In the Magdalena range, near the town of Kelly, blende occurs. The blende is dark and contains considerable amounts of iron. It will analyze about 19 per cent zinc and 4 per cent to 6 per cent lead and is concentrated to an ore of 45 per cent zinc. A large part of this ore is used for the manufacture of zinc oxide.

Montana produces zinc principally around Butte. The Montana ores come mainly from mines formerly operated as silver producers but abandoned because of the low price of silver. The Black Rock Mine is the leading producer. The ore will analyze about 16 per cent zinc. It has a low lead content, and can be concentrated to about 52 per cent zinc. Zinc also occurs as sphalerite (the sulphide) associated with pyrite, rhodenite and rhodochrosite. Galena is found very sparingly.

Idaho, in the Coeur d'Alene district, produces blende and blende with galena. The ores are rich sulphides

and are silver bearing. They are subjected to concentration and the zinc is recovered by electrolysis.

Utah has been a producer of blende for many years and recently produced oxide ores. There are ore bodies in the Park City region which carry silver to the amount of 10 to 25 ounces a ton in concentrates, and 40 per cent to 45 per cent zinc. Ores are also found in the Tintic district.

Arizona has its principal zinc producers in Mohave County. The ore is dark blende, carrying considerable amounts of iron, also carrying galena. The Tennessee mine concentrates will analyze about 40 per cent zinc.

Nevada produces zinc ores in the Yellow Pine district of Clark County. The minerals of this district are zinc carbonate and silicate. The ores analyze about 32 per cent zinc, 10 per cent lead and 5 ounces of silver a ton. They are concentrated to about 40 per cent zinc and 12 per cent lead and are shipped to zinc oxide plants.

California has blende with sulphide copper ore in Shasta County. The crude ore contains 30 per cent to 35 per cent zinc. In Inyo County oxide ores with galena occur as replacement of limestone.

CHAPTER 3

ZINC OXIDE

Zinc oxide is a white, snowy, metallic pigment, in all appearances resembling household white flour. It is formed by vaporizing zinc, either directly from the ore or "indirectly" from spelter (slab zinc) and allowing this vapor to come in contact with an oxidizing atmosphere. The vaporous zinc really "burns" in air and as a result it produces an intense flame and the "white smoke" of zinc oxide forms. In commercial plants, the zinc oxide laden air is drawn through cooling pipes and the pigment is later caught in the meshes of muslin bags. The manufacture of zinc oxide directly from the ore is known as the American Process, and the manufacture of zinc oxide from spelter is known as the French Process. Both processes shall be described later.

Zinc is volatile or will become vaporous at a temperature of 1000° C., the boiling-point of zinc being 920° C. The chart of Doeltz indicates that volatilization is rapid at 1300° C. and practically complete at temperatures of 1370° C. and 1400° C. The dissociation temperature into Zn and O has been calculated by Stahl to be 3817° C. The working temperature of furnaces is usually about 1100° C., as the reduction temperature varies with the character of the ores and the zinc oxide to be produced, as well as the reducing carbon—some ores being easier to reduce than others.

Zinc oxide is insoluble in water, will not change its color when exposed to temperatures ordinarily met in commercial uses of this pigment, and is unaffected by light, cold or darkness—or wind, rain, and gases found in rather concentrated quantities about industrial centers.

Zinc oxide has a very fine particle size, being the finest paint pigment in common use, and for rubber

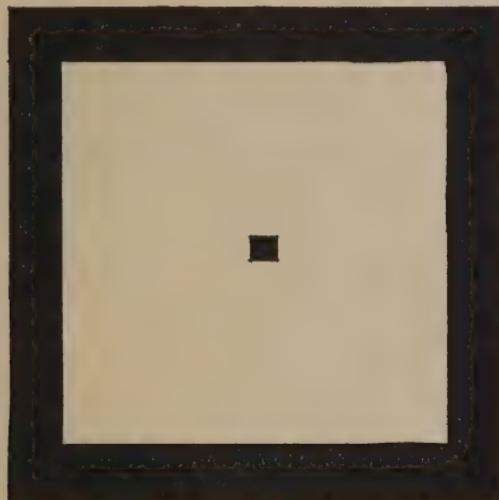


FIG. 3.—Relative size of zinc oxide particle and the space of a 300-mesh screen.

compounding it is considered second to carbon black in fineness. It has such a fine particle size that the highest powered microscope must be used in order to see any of the outline of the individual pieces. The relative proportion of a particle of zinc oxide can be shown by representing it as a small square in the center of a magnified space between the wires of a 300-mesh screen.

Upon looking through the microscope at different pigments the comparative size of the particles can easily be seen, and it will become evident that zinc oxide varies considerably from the other white pigments, and even the different zinc oxides will vary as against similar pigments produced by a different process.



FIGURE 4.—Photomicrograph of French Process Zinc Oxide
(3000 Diameters)

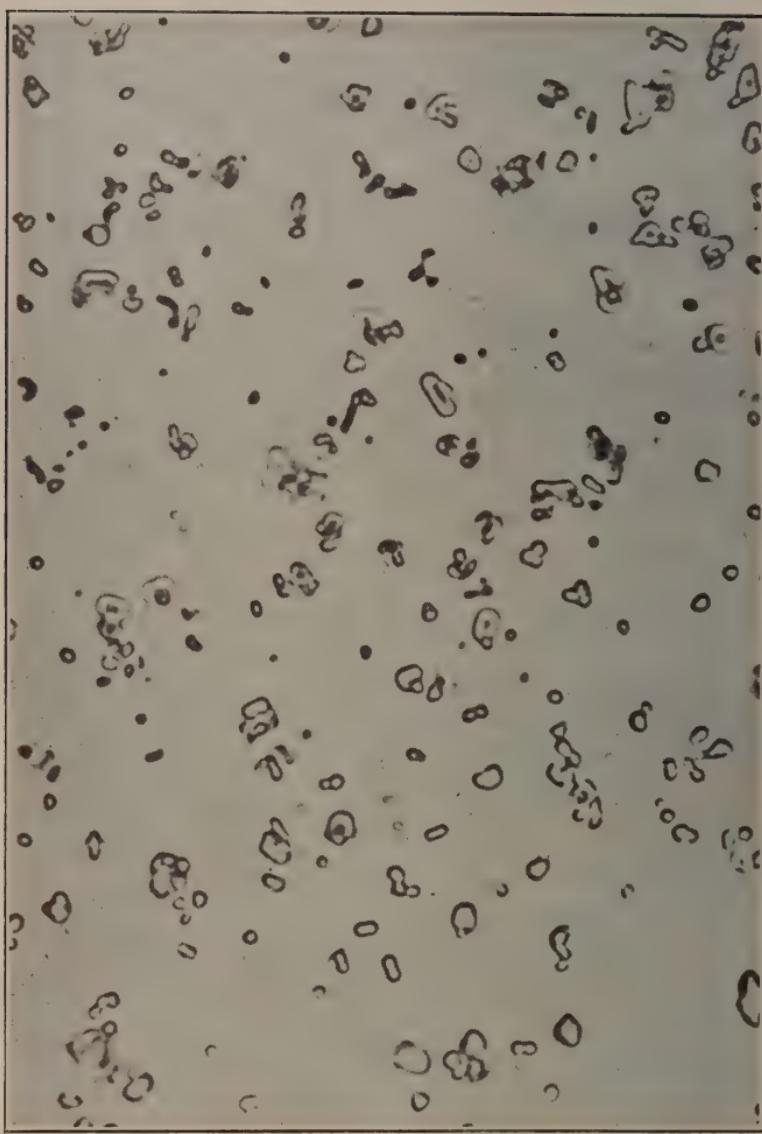


FIGURE 5.—Photomicrograph of American Process Zinc Oxide
(3000 Diameters)

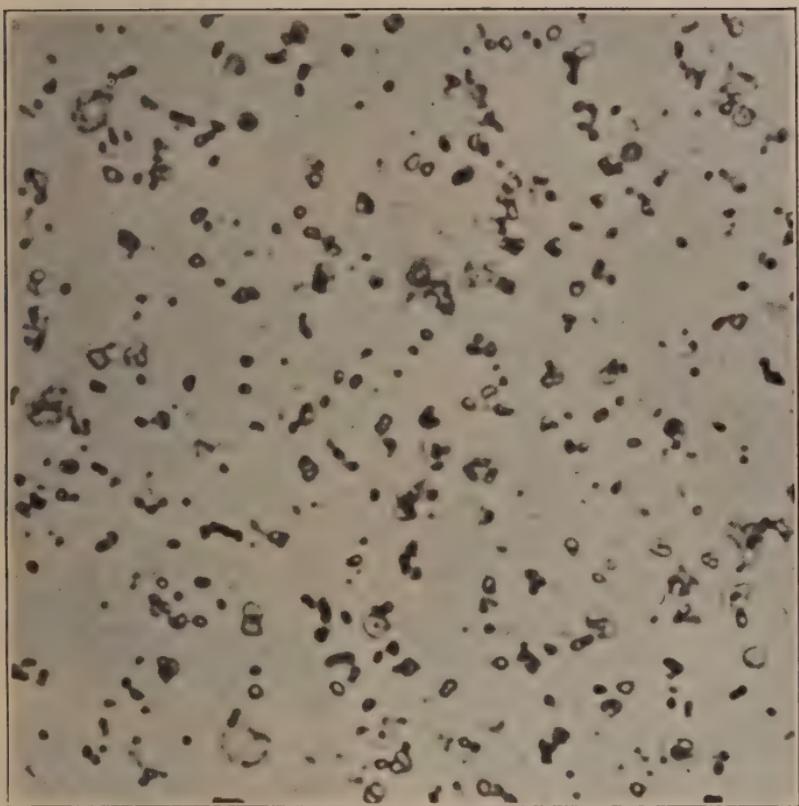


FIGURE 6.—Photomicrograph of Lithophone (3000 Diameters)

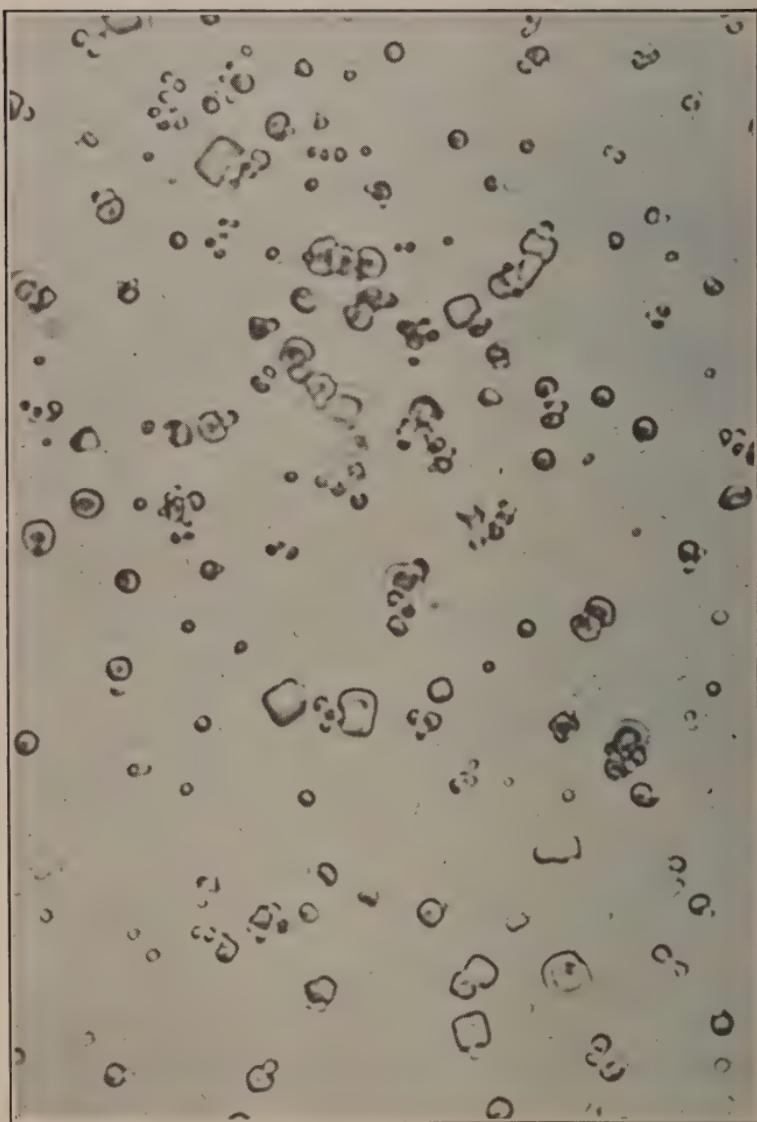


FIGURE 7.—Photomicrograph of Sublimed White Lead
(3000 Diameters)



FIGURE 8.—Photomicrograph of Corroded White Lead
(3000 Diameters)

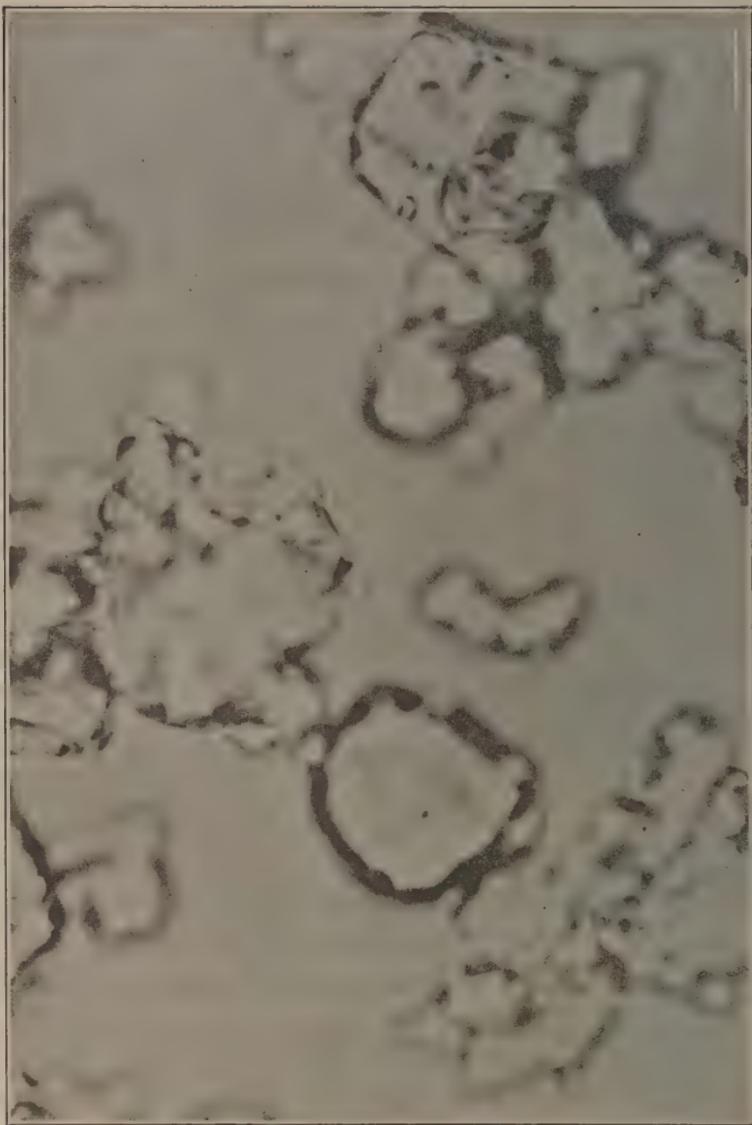


FIGURE 9.—Photomicrograph of Barytes (3000 Diameters)

CHAPTER 4

FRENCH PROCESS ZINC OXIDE

The Manufacture of Spelter

The French Process, or the "indirect process" of manufacturing zinc oxide was first used in France by LeClair and Sorel during the latter part of the eighteenth century. In this process the zinc is reduced from the ores to a metallic state. The spelter, or slab zinc, is then used in the second step by being vaporized in a specially constructed furnace and the volatilized zinc allowed to come in contact with an oxidizing atmosphere. The zinc oxide thus formed is cooled and collected by means of a baffle plate chamber and later muslin bags. Inasmuch as the manufacturing of spelter or slab zinc is necessary for the first step in the production of French Process zinc oxide it shall be briefly described.

Spelter, or metallic zinc, is produced from zinc ore by the process of distillation. The zinc ore is mixed with coal, charged into a cylindrical clay retort, heated to 1200° C., where the zinc is vaporized and allowed to cool to about 600° C. and condense to the metal in a separate clay "condenser." The metal is drawn from the condensers while in a fluid state and cast into slabs, where it cools, crystallizes and becomes the commercial product—spelter.

The furnace used for smelting zinc ores is composed of two sections, the base or the heat regenerating chamber is one section and the laboratory or the upper half of the furnace is the other section. In the laboratory are suspended the retorts used in the smelting of zinc bearing ore.

There are many different types of furnaces used for the smelting of zinc, such as the Belgium, Carinthian, English, Rhenish, Silesian, and many types bearing the names of their designers. In principle they are the same. In order to demonstrate the process and to give the reader some conception of the process and principles involved a brief description of the Convers and De-Saulles Furnace, used in this country, shall be given.

The lower section of the drawing can be seen to be the base or preheating section of the furnace. This pre-heating section has a height of 14 feet 6 inches, it is 17 feet 6 inches wide and has a length of 35 feet 4 inches. It is constructed of heavy fire brick and masonry, and it is so arranged that it has two chambers containing checker work of brick. A series of gas and air inlets and outlets are arranged to conduct air and gas to and from the upper section, called the "laboratory" where combustion takes place.

The operation of the pre-heater is solely for the purpose of pre-heating air before it is led to the laboratory. Air is forced through one side of the brick checker work (which is very hot) and in coming in contact with the hot bricks it absorbs heat. It is then led to a distributing main and allowed to enter the laboratory of the furnace through an opening at the base of the center wall of the laboratory. It is then distributed to a series of outlet ports through which it escapes to

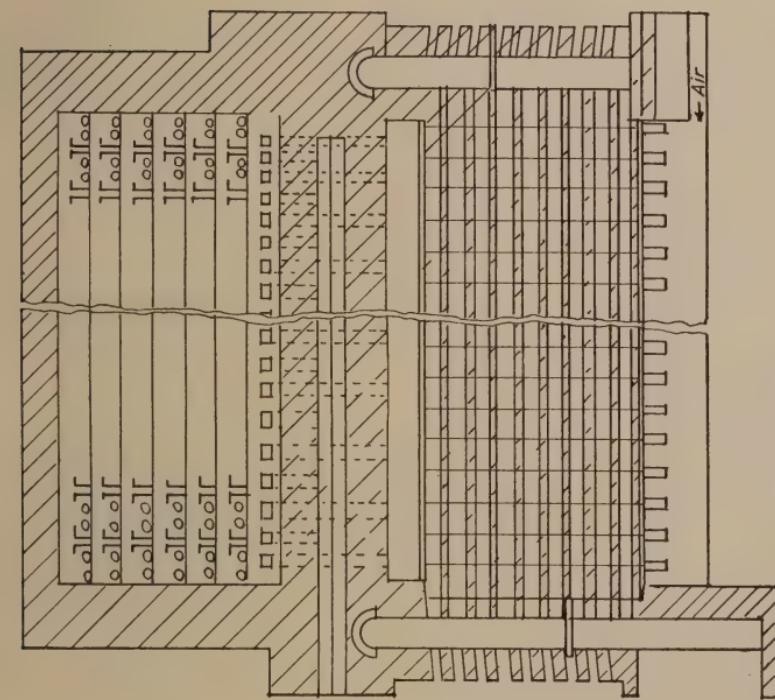
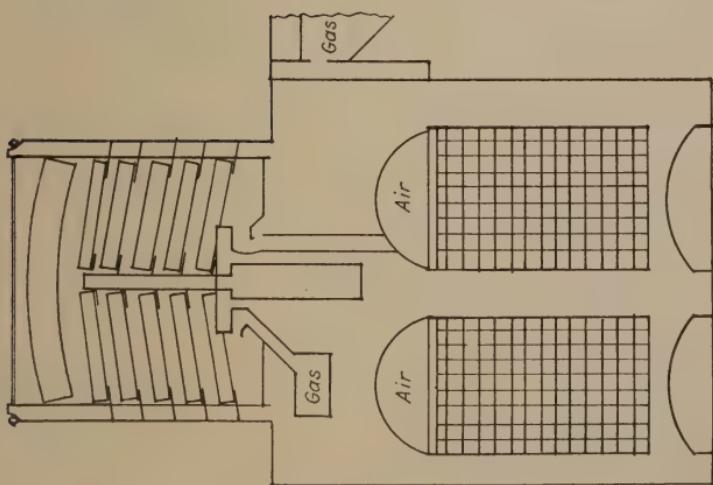


FIG. 10.—Convers-DeSaulles zinc smelting furnace.



the combustion section of the laboratory. Gas is led through a separate main and sent through distributing sections to laboratory outlets just above the air ports. Combustion takes place and the gas is burned to furnish a temperature of about 1200° C. to 1300° C. necessary for the smelting of the furnace charges. The burned gases are led from the laboratory through ports in the center wall on the opposite side from the feeding ports, and conducted through a separate passage to the second section of brick checker work in the base of the furnace. The brick checker work absorbs heat from the gases and soon becomes very hot. From this chamber the gases are led to a stack and allowed to escape to the air. During this procedure the one section of the recuperating checker work is being cooled by the ingoing air and the other is being heated by the spent gases, so that the procedure of reversing the flow of the burned gases and air every fifteen minutes permits the recuperation of waste heat of the burned gases and increases the efficiency of the fuel, by preheating the air.

The gas consumed in the furnaces is made from coal by the use of gas producers. There are also several types of gas producers, such as the Hegler, the Chapman and the Hughes. They are upright, cylindrical, brick-lined sheet-steel retorts having a revolving body the ends of which are water sealed into a top and a bottom section. At the top the revolving jacket is sealed into a specially constructed iron cover, which has the feeding hopper through which ground soft coal is fed, poke holes allowing the use of bars to break up any clinkering on the inside of the producer, and the gas lead off. The bottom of the revolving jacket is water sealed

into a funnel-like section for the collection and removal of ashes. Just above the funnel-like bottom there is a hearth upon which rests a bed of hot coals and through which air and steam are blown.

The air and steam being forced through the bed of hot coals oxidizes some of the coal and causes chemical changes in the upper section of the producer. The reaction of $2\text{H}_2\text{O} = 2\text{H}_2 - \text{O}_2 - \text{C} - \text{CO}$ takes place. The carbon dioxide that forms passes through the coals and becomes carbon and carbon monoxide. The main gas stream led from the producers also contains oxygen and methane. The producer is designed to produce carbon monoxide gas but actual analysis of the gas shows the CO to be only about 25 per cent to 30 per cent of the total. The gas also contains about 4 per cent CO_2 , 12 per cent H_2 , and 57 per cent N.

The laboratory or the upper section of the spelter furnace is composed of a firebrick shelf section built up through the center. This section serves to support the one end of the retorts as well as the center of the arch over the top of the furnace. The sides of the laboratory are constructed of cast-iron frames and firebrick, having the frames anchored at the base of the furnace and bound together by rods across the top. The arched ceiling of the laboratory is supported both by the bound sides and the center wall.

Retorts are suspended in the laboratory, the base or the closed end of the retort being set on the shelves of the brickwork in the center wall and the open end being supported by the front of the furnace. These retorts are arranged in rows and tiers on both sides of the furnace, having 120 or 130 retorts to a side, or 240 or 260 to a furnace.

The retorts are oval in shape and made of fire clay. They are 57 inches long inside and 9 inches high and 7 inches wide and have a volume of 1.62 cubic feet. They are made in a special refractories plant from bond clay, grog, etc. The grog used is composed mainly of pre-used materials such as broken saggers, brickbats,

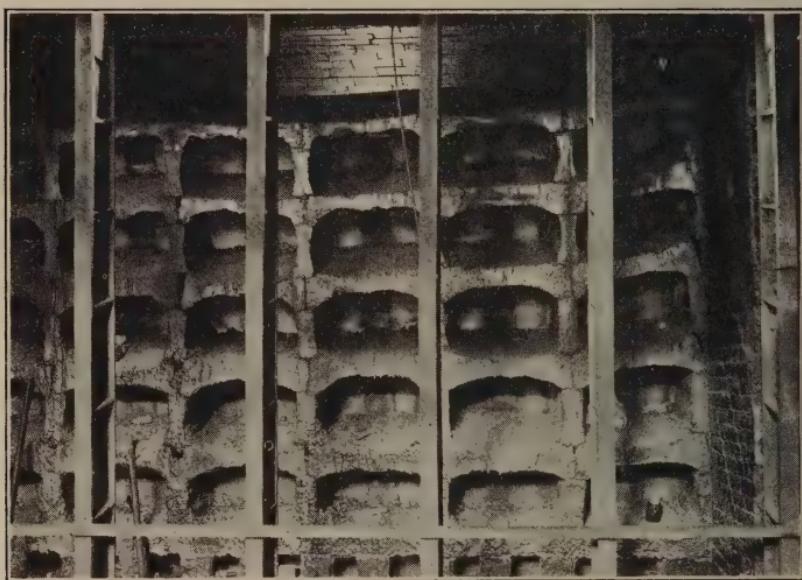


FIG. 11.—View of the inside of a zinc smelting furnace. Note the center section, the air and gas ports, the arched ceiling, and the side frames.

etc. The material is crushed, screened, put through pug mills for thorough kneading, aged, re-pugged and moulded into retorts by large hydraulic pressing machines. These vessels are dried for a period to free them of as much moisture as possible, later brought to the furnace floor where they are placed in a baking chamber for the purpose of driving off any chemically combined water and to strengthen them so that they

can withstand the intense heat of the laboratory, and are brought to a red heat so as to be ready for use in case any replacements are necessary for the breakages in the furnaces. The retorts are set in the laboratory by means of bars and are so arranged that the base or closed end is 4 inches above the level of the fore or open end.

Another important part of the furnace is the condenser, a bottle-like clay cone which is set at the open end of the retort to collect, cool and condense the zinc vapors to metallic zinc. The condensers used are about 2 feet long, have $7\frac{3}{4}$ inches O.D. at the base and $4\frac{1}{2}$ inches O.D. at the other end.

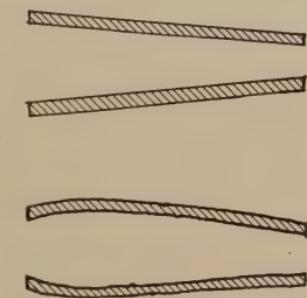


FIG. 12.—Zinc furnace condenser.

Condensers are made of the same clay as are retorts, are moulded by machine, have the base crimped so as to fit into the mouth of the retort and are burned at a temperature of 900° C. There is a condenser for each retort, and they are suspended in the open air by a hinged iron bracket attached to the side of the cast-iron furnace frames.

The raw material, or charge, used for the production of spelter consists of zinc ore in proportion of 18,000 pounds, dust coal 7000 pounds and common salt 40 pounds. This charge is churned to an even mixture in a Ransome concrete type of mixer, taken by a special skip to a storage bin, from which it is later drawn into

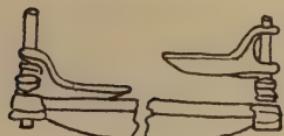


FIG. 13.—Condenser support.

charge larries which carry it to the furnace floors. The charging car, on which the larries deposit the charge, is a flat table-like platform, having four wheels permitting it to be rolled forward from the back wall of the furnace building to the face of the furnace at the time it is desired to charge the retorts.

The discharging and recharging, as well as the entire operation of the furnace, are carried on by manual labor and as the filling and handling of the retorts must be done individually by laborers, the operators are subjected to intense heat during the time of this laborious task.

A charge crew of six men remove the hot condensers from the face of the furnace by means of condenser rods.

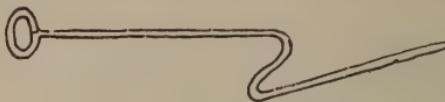


FIG. 14.—Condenser rod.

The condensers are set aside and when cool will be cleaned by a member of the crew. Droppings and the old ore-coal mixture of the former charge are scraped from the front of the retort, and saved for a later recharging. A sheet-metal shield is dropped over the front of the furnace and under its protection a member of the crew inserts a long pipe into the retort, and by means of a hose attachment to the pipe he introduces water into the back end of the red-hot retort and by the formation of steam at that point in the retort the old charge is "blown out."

This is done to all of the retorts. The refuse drops to the floor and then through hopper openings in the floor at the side of the furnaces. Further cleaning of

the retorts is then done by chisels if necessary. Breakages are removed by means of hand bars and new retorts are inserted. After the furnace is discharged and the floor is cleaned the charging table is rolled to the front of the furnace and the men proceed to charge the retorts.

The mixture of coal, ore and salt is thrown into the retorts by means of a special scoop shovel. The men throw the charge into the retort until it becomes full and then run a rod through the top of the charge to

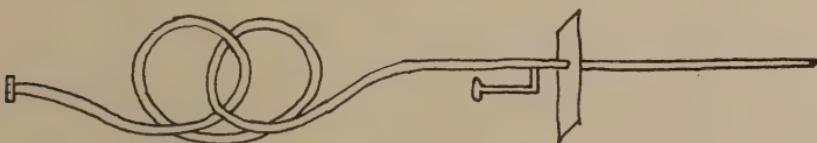


FIG. 15.—Blowout pipe and hose.

permit the outflow of gas and prevent a blowout. It takes three and one-half hours to discharge and recharge a furnace.

The retorts being filled the condensers are then placed. They are placed by forcing the crimped larger end of the condenser into the open end of the retort and then resting the neck or fore part of the condenser on the "grasshopper arms" or brackets which protrude from the front of the furnace. The openings of the condensers are luted or sealed shut by moist mixture of coal and ore in order to seal off any outside air but at the same time to allow internal gases to escape. Carbon monoxide gas is generated inside the retort and does escape through this luted mouth of the condenser. Being a poisonous gas it is desirable that it be destroyed and for this reason the gas is burned at the mouth of the condenser. In burning with a bright yellowish

flame the gas causes each condenser to appear as a lighted torch, giving the furnace block, with 120 or 130 flaming condensers to each side, a very unique appearance, and especially so at night.



FIG. 16.—Zinc furnace in operation. Note the condensers and the burning gases issuing from them.

The action of the charge within the retort varies directly with the temperature. The reduction of zinc

from the ore, by the presence of carbon, does not begin until the vessel has reached the temperature of 900° C. to 950° C. Thus, during the "warming up" period, the carbon has little effect upon the reduction of metallic zinc. However, below the temperature of 800° C. there will be a reduction of zinc due to methane, CO_2 formed in the charge and hydrogen. The CO_2 being abundant acts as an oxidizing agent upon the zinc vapors and causes a formation of crude zinc oxide and blue powder, commonly known as zinc dust. As the heat of the charge increases the relative amounts of CO_2 will decrease and CO increase in proportion. The zinc is vaporized in the retort and comes to the condenser where it is cooled to 600° C., crystallizes or condenses and forms metallic zinc.

The furnaces are charged once in twenty-four hours, the hours of charging being usually four to seven-thirty in the morning. Draws of the metal are made three times a day, usually at 2 P.M., 10 P.M., and at 3 A.M. The zinc is removed from the condenser by inserting a scratcher button, which is a rod having a blunt end, and drawing the molten zinc into a suspended movable kettle, which is swung under the condensers. The drawn metal is poured into a mixing pot and later cast into slabs.

Slab zinc, commercially called spelter, is used principally for galvanizing sheet steel and for brass manufacturing. A portion of the natural production of spelter is rolled into sheets and strips for roofing, etc., and still a smaller percentage is used for the manufacturing of French Process Zinc Oxide.

CHAPTER 5

FRENCH PROCESS ZINC OXIDE

The Manufacture of Zinc Oxide

The furnace used in manufacturing French Process zinc oxide very much resembles a spelter furnace, as it also has a recuperating pre-heating base and a laboratory in which are suspended a row of retorts very similar to the retorts used in the manufacture of spelter.

There are four brick checkerwork recuperating sections in the base of the furnace, two of which are used for gas and the remaining two are used for air. One of each set is used at a time for pre-heating, the other one being heated up in the meantime by the outward passage of burned gases. The flow of the gas and the air is reversed every half hour so that the ingoing gas and air may be maintained at a high temperature.

The upper section of the furnace is a brick encased combustion chamber, across which is suspended a row of muffles. The gases coming from the preheating section burn in this combustion chamber giving an intense heat around the muffles and causing a constant temperature of about 1400° C. The gases, after burning, pass down from the combustion chamber to the recuperating brickwork in the base and then out to a stack. Above the combustion chamber is a separate brick compartment known as the gas chamber. Into

this chamber, which is merely a brick rectangular space, is fed carbon monoxide gas, which was generated in a producer and cleaned in a scrubber beneath the furnace floor level. The carbon monoxide gas is heated

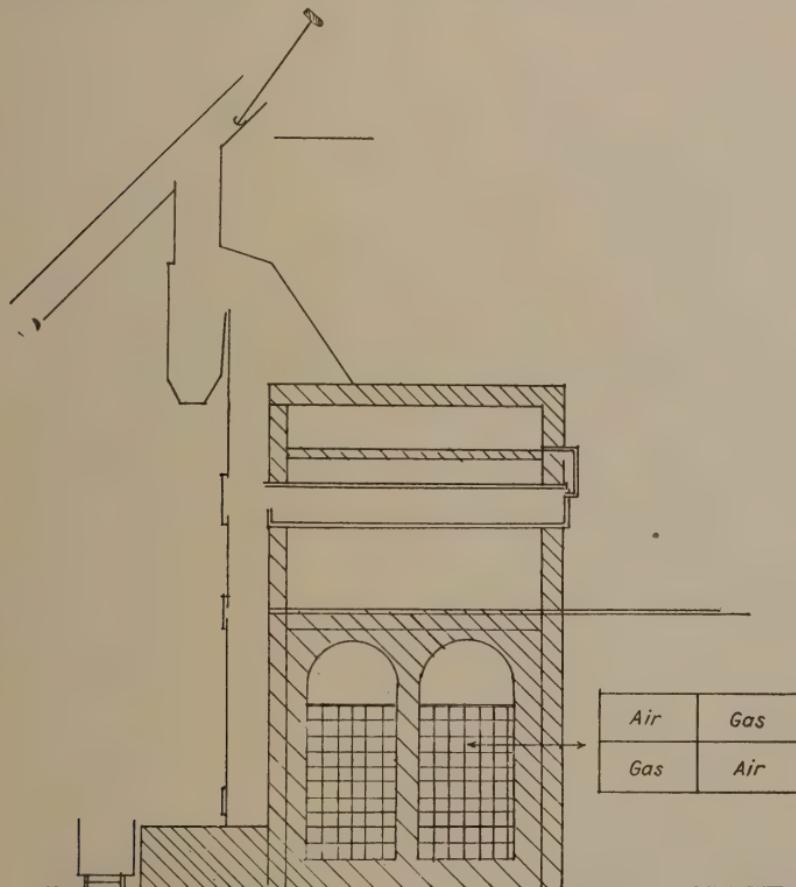


FIG. 17.—French process zinc oxide furnace.

by the warmth of the firebrick partition which is between it and the combustion chamber. It is removed from the gas chamber by means of a pipe leading from an opening in the chamber to the charge end of a muffle.

The muffle or retort used in the French Process zinc furnace is the same size and shape as a spelter retort. The one end is open while the other end has a dam, or half-closed face. The muffle is suspended so that the open end extends to the outer surface of the rear supporting wall of the furnace, and the dam end extends to the outer surface of the front supporting wall.

The fore end of the furnace has a sheet-iron section running the entire height of the furnace. It has three doors for each section of muffles, one at the level of the muffles, one at the level of the base of the laboratory and one at the bottom of the furnace. Above the sheet-iron section or chamber is a collection pipe, being the broad side of a cone which leads to a collection sector above.

The muffles of the furnace are charged with spelter. The slabs of zinc are chopped or broken up into convenient sizes and placed in the muffle through the open end at the rear of the furnace. A small portion of clay is also thrown into the muffle to prevent the metal and slag from clinkering to the sides of the muffle at the time of cleaning. The usual charge consists of two or three parts of high-grade spelter to one or two parts of Prime Western (higher leaded) spelter.

The muffle is not completely filled with spelter, but a space of about 3 inches is allowed at the top of the muffle for vaporization. After the spelter has been placed in the muffle a pipe set in the shape of a U is inserted in the gas chamber through an opening in line with the muffle. This is done so that a stream of carbon monoxide gas may be led to the muffle and serve two purposes, namely, prevent oxidation in the muffle and to lead the zinc vapor to the mouth of the muffle.

After the pipe is set in the muffle opening the back of the muffle is then closed with clay, the plastering being made to conform to the pipe and made so as to seal the end of the muffle. The clay is tamped and made secure in place by means of a red-hot blunt-ended rod.

The zinc contained in the muffle soon melts and as the temperature rises it vaporizes. The zinc vapor is induced to flow to the front end of the muffle by the stream of carbon monoxide gas and there it comes in contact with air or an oxidizing atmosphere and "burns" with a bright yellowish flame to zinc oxide. The front end of the muffle is the only means for checking the contents of the muffle. As soon as the flow of zinc oxide begins to decrease it is obvious that the charge is becoming exhausted.

After the charge of spelter has been fairly well reduced and the amount of the zinc in the muffle is getting low, a second charge is made. This charge is of molten metal which has been brought to the side of the furnace in a ladle. A slide, placed in the end of the gas pipe, is opened and a tray-like funnel is inserted. The molten metal is thus poured into the muffle and the proper amount of zinc is charged. These molten charges are made as often as are necessary. It is arranged, however, so that the muffles can be opened and cleaned once in forty-eight hours.

The cleaning of the muffles is done in order to remove any of the impurities which form in the muffle and cause a decrease in the production. The charging end of the muffle is chiseled open and all of the clay removed. The gas pipe is removed and disconnected from the gas chamber. The muffle is then scraped clean by means of a spoon-like rod which reaches to the

other end of the muffle and draws all refuse material back with it. There may also be a clinker formed at the side of the muffle which will necessitate chiseling in order to remove it. The refuse from the muffle is dumped on the charge floor, allowed to cool and then stored for shipment. This refuse contains about 40 per cent zinc and 20 per cent lead and can be used in the manufacture of leaded zinc oxides.

There are many breakages of muffles due to the deterioration from the charge, from the intense heat, and from the wear and tear caused by cleaning. A furnace usually has twenty muffles and has about eighty replacements a month. Zinc saturated sections of broken muffles are recovered and used for spelter or zinc oxide charges.

The stream of zinc vapor pours from the open end of the muffle and there combines with the oxygen of the air and forms zinc oxide. The stream of zinc oxide, now appearing as a dense snow floated in air, is drawn up from the furnace to a collection main and led to the main collection hopper. A small portion of the zinc oxide falls to the catch hoppers at the base of the collection main, from where it is removed, screened and used in the main products of the plant.

Accretions, known as horns, form at the fore end of the muffle. These horns, or hardened and fused accumulations of zinc oxide, act as condensers to the zinc vapor coming from the muffle and may cause the vapor to remetallize and form metallic zinc as a coating. In order to stop this action a man is located at the base of the iron section in front of the furnace and by means of a long rod having a scraper he keeps the face of the muffle scraped clean of oxide horns. Any horns that

form are scraped off and fall to the base of the sheet-iron section and are then collected at the base floor level.

From this accumulation of horns and zinc oxide scraped from the muffle face is reclaimed some rough oxide. This is done by having the accumulated scrapings dumped into a large screen where the heavy horns are removed. These horns are segregated, barreled and are later used in the manufacture of another white pigment, lithopone. The zinc oxide passing through the screen falls to the base of an elevator chain which carries it to a trommel above. Here all of the fine horns and heavy particles are removed. The zinc oxide is allowed to fall into a blowing section, and the blast carries the light oxide from this cleaning station to a bag collection unit. All of the refuse or heavy zinc oxide and horns collected in this cleaning process is barreled and later shipped away for the use in making lithopone.

To return to the furnace—the main stream of zinc oxide is led through a large sheet iron pipe to a large collection hopper. The collection hopper is a large oblong sheet metal chamber with baffle plates extending from both top and bottom. The baffle plates are so alternated that the incoming zinc oxide streams must follow a zigzag course, hitting the plates and the sides of the collection chamber at every turn. The heavier zinc oxide falls first and the lighter zinc oxide is carried to the further end of the hopper. The zinc oxide that falls to the bottom of the collection chamber is removed by means of hopper doors, as the bottom of the chamber is arranged as a series of small hoppers. A lead off pipe at the far end of the hopper allows the

zinc oxide still remaining in suspension to be led to the secondary collecting unit.

Leaving the collection chamber the air, still heavily laden with zinc oxide in suspension, is fed to a blow fan—the agency of action for the stream—and from it is forced into collection bags. The bags used are made of muslin and are about 3 feet in diameter and hang

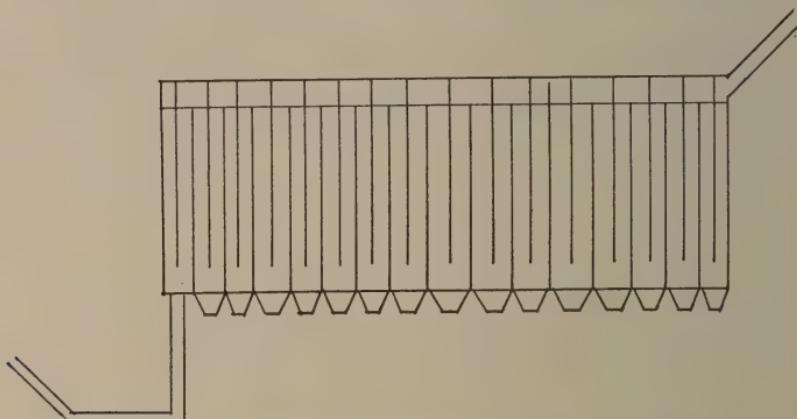


FIG. 18.—Collection chamber. Showing baffle plates and hoppered bottom.

20 feet long. They are attached at the top to round outlets in the iron pipes and are kept closed at the bottom by strings or knots. The interstices of the collection bag's muslin allow the air and gases to escape but imprison the zinc oxide particles. The zinc oxide clings to the side of the bag, is shaken loose periodically and is caught at the bottom of the bag where it is collected and taken in bags to the mixing room.

The zinc oxide that collects at the further end of the collection chamber and at the bags is always the lightest and fluffiest and finest in particle size. It contains a large amount of "imprisoned" air and is consequently

lighter in specific gravity and less bulky than the other zinc oxides. It is also very clean and free from dirt. This being the first grade zinc oxide it is classified as White Seal. It is blended to assure conformity to a standard sample, bolted through a flour bolter to have it thoroughly cleaned of any fibers or foreign matter, and is then packed in 150-pound barrels.

The zinc oxide collected at the central section of the collection chamber is more dense, and the zinc oxide collected at the first part of the collection chamber is the heaviest of all. It will be noted that there is no chemical difference in the gradings of this zinc oxide, but the grades of French Process zinc oxide differ solely in their physical properties, such as gravity and color. If cadmium is present in large degrees in the spelter of duplication the charge it will cause the zinc oxide to appear yellow.

The zinc oxides, classed as Green Seal and Red Seal, taken from the collection chamber are then subjected to a special heat treatment to give them a denser body. The collected pigment is elevated to a platform and allowed to fall through a superheated brick vertical muffle.

The muffle used in the reheating of the zinc oxide is made of brick, having a height of 30 feet, and has a 15-inch square opening running the entire height, the opening being divided into three sections by two brick dividing walls. The muffle walls are heated by being encased in a fire-box. The zinc oxide is poured into a hopper at the top of the muffle and from it is fed by a revolving cylinder grate so that the amount going down the muffle is not greater than the temperature of the muffle can care for. The zinc oxide falls like snow and as it

falls is heated. The heat causes the zinc oxide to become compact and frees it of a large percentage of the imprisoned air. At the bottom of the muffle the zinc oxide falls, red hot, on a wide flat pan-like truck. The truck is so devised that the zinc oxide cools readily. As soon as it is cooled it is taken to the mixing bins and there allowed to cool to the normal temperature. It is blended to meet a standard sample, bolted and packed into 50-pound bags or 300-pound barrels.

French Process zinc oxide is used principally for making white and tinted enamel paints. Its uniform very fine particle size and good color especially recommend it for this use. It is also used to some extent in outside white paints. The pottery manufacturers use it in the compounding of their vitreous glazes. Manufacturers of cosmetics also use this grade of zinc oxide, generally White Seal, for face powders. Recently rubber manufacturers have been using large tonnages of this grade of zinc oxide in rubber compounding.

It is generally agreed that the best French Process zinc oxide of the world is made in the United States. Imported French Process zinc oxides vary chemically and physically, especially in sulphur content, while American production is standard and uniform. Great developments may be anticipated in the American production of this product, principally because of the exacting demands of the rubber manufacturers and the severe competition that other mineral pigments are giving for this use.

Pharmaceutical Zinc Oxide

Pharmaceutical zinc oxide is made by the French Process. A few variations in the actual operation of a

plant are made because U. S. P. zinc oxide is the best zinc oxide made both chemically and physically and every possible precaution is taken to assure purity.

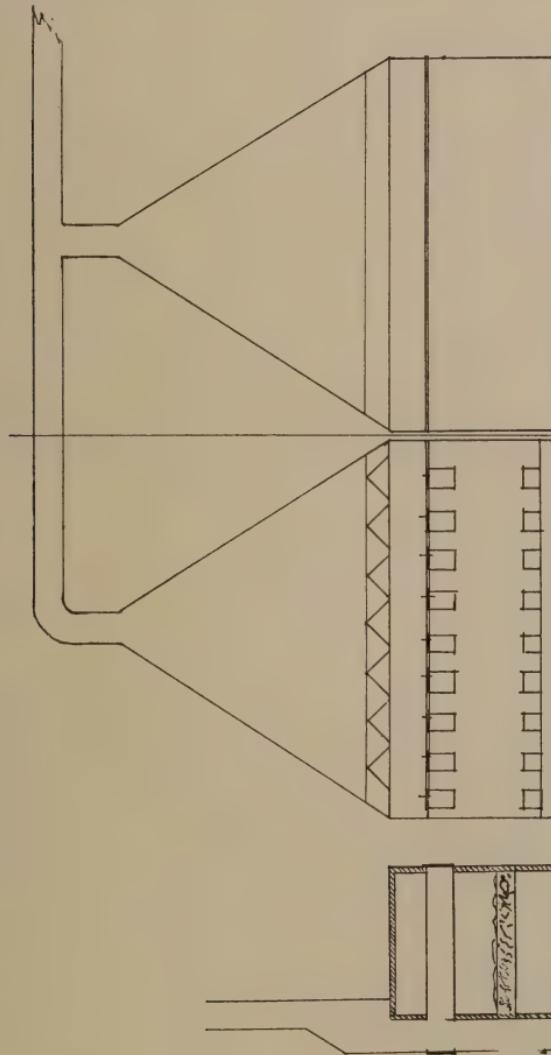


FIG. 19.—Pharmaceutical Zinc Oxide Furnace.

The furnace used to produce this grade of zinc oxide does not have a recuperating section, and does not use gas as fuel, but is heated to a temperature of about

1200° C. by means of two coal-fed fire-boxes placed at each end of the brick furnace structure. Thus the furnace very much resembles a fire-box, fed fuel at both ends and having a row of muffles or retorts extending side to side and placed near the top of the furnace.

The muffles are similar to the retorts or muffles used in the making of spelter or French Process zinc oxide, having a capacity of 1.28 cubic feet of molten zinc. The muffles are kept at a constant temperature, being fed a "stick" of spelter every ten minutes. These sticks of spelter are especially selected for this use because of chemical composition and are cast so as to facilitate charging of the muffle.

The zinc oxide which forms at the mouth of the muffle is drawn up through a collection downcomer, to a fan and forced from there to a bag house where it is collected in the meshes of muslin bags. It is packed directly from the collection bags to well-lined barrels. Only 100 pounds of U. S. P. zinc oxide are packed loosely into a large barrel.

Pharmaceutical zinc oxide is used exclusively for medical purposes—such as zinc ointment, healing powders, etc. It has a very high antiseptic value. It is also used in some of the higher grades of face powders.

CHAPTER 6

AMERICAN PROCESS ZINC OXIDE

The American Process, or direct process, of manufacturing zinc oxide has been used since 1852. It is called the American Process because it was invented and developed in this country. The principle of the process is to vaporize the zinc directly from the ore, the reduction being facilitated by carbon, and then allowing the zinc vapor to oxidize. In the process a mixture of zinc ore and coal is spread on a body of burning coal, which, in turn is on a perforated grate, and an excess of air is blown through the grate. The heat of the burning coal and the reducing action of the CO gas causes the zinc to volatilize and it burns to zinc oxide by the combination with the excess of air in the upper part of the furnace, and in the lead off flues. The zinc oxide with all of the products of combustion from the furnace, and the excess of air, is carried to a bag room through a carrying system of pipes which also act as cooling agents. In the bag room the gases escape through the meshes of the muslin bags, while the zinc oxide becomes entangled and is screened out or imprisoned in the bags.

There are two distinct types of furnaces used for producing zinc oxide by the American Process, known as the Eastern Type and the Western Type. There are also combinations and variations of the two, as

well as several new types of furnaces that represent indicative trends for the future. The largest and newest plant producing zinc oxide by the American Process is located in the East, about one hundred miles inland from New York City. A description of the plant and an outline of the procedure at this plant will give the reader a clear understanding of the American Process as practiced in the East.

The general plan of this Eastern zinc oxide plant—the largest in the world—is shown in the accompanying drawings. It can be seen that the principal sections of the plant are the storage yard, for the ore and coal, the stock bins and mixer, furnace, fan house and bag room. These individual parts of the plant shall be discussed separately and we shall endeavor to trace the complete journey of zinc from its reception at the plant in the form of raw ore to the departure of the finished product in the form of zinc oxide packed in the car.

Zinc ores and anthracite coal coming to the plant are brought up an incline to a two-track unloading and storage trestle. The trestle is a long concrete structure, being sufficiently wide to accommodate two tracks. The incoming cars are propelled by an electric truck car and are placed for proper unloading. A concrete wall runs through the middle of the trestle and to its entire length. This is done so that the track on the furnace side can empty cars directly into steel bins for current needs while the track on the opposite or the storage side of the trestle can empty the contents of the cars to an open platform for removal to the outside storage piles.

The trestle also incorporates a concrete foundation and a single rail for one set of wheels of a large 233-foot

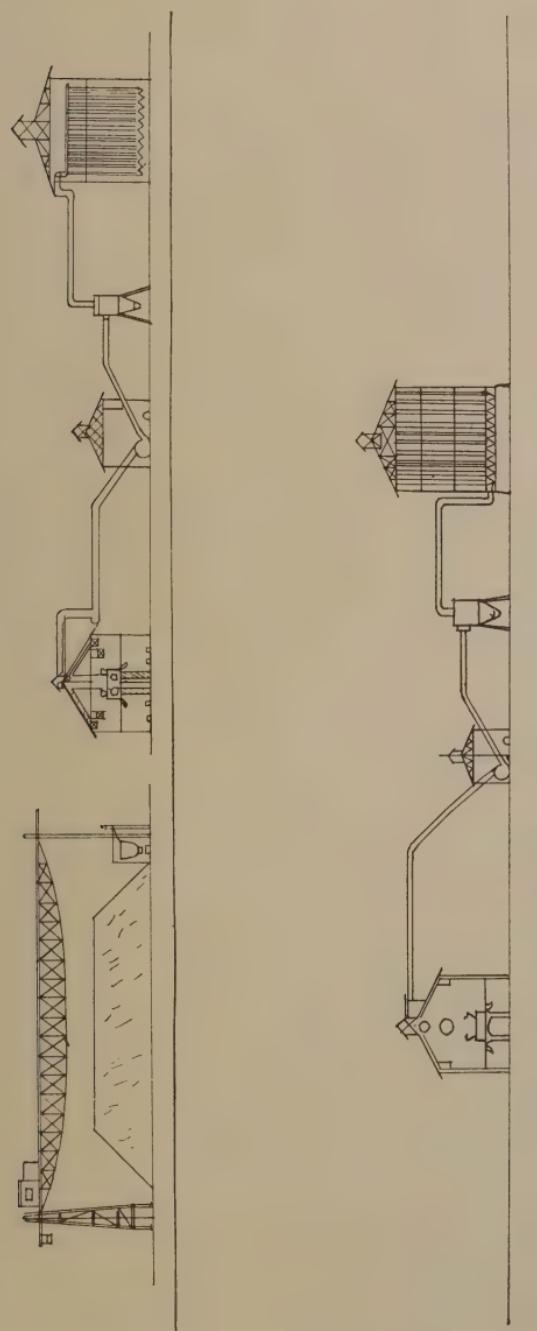


Fig. 20.—Cross-section view of zinc oxide plant at Palmerton, Pa.

gantry crane. The crane has a 5-ton bucket and a working radius of several hundred feet. Ores and coals for long storage are piled by the crane, and later, when needed, are brought back to the trestle by means of the crane and this time placed in the current material bins. It is generally considered good practice to store some ores for a period as they need weathering, and by this weathering they become more uniform as to their moisture content.

Current materials are taken directly from the cars and fall to bins on the furnace side of the trestle. The cars are all unloaded by opening the bottom hoppers and allowing the material to fall through the iron gratings into Brown tangential bins. As the materials are unloaded they are sprayed with water from above the car. This is done in order to have the materials well soaked, which permits easy handling and is desirable for cohesion of the charge in the furnaces. In cold weather this watering is done away with as snow and general weathering give sufficient moisture. Steam is generally used to melt the frozen ores and coal and this also imparts moisture to the materials.

Samples of all incoming materials are taken from the car at the time of unloading. The samples are taken according to standardized methods, giving a representative average of the car. These samples are then taken to a special sample house, a brick and steel building, equipped with driers; compact, rotary and grinding types of crushers and bottling facilities. The samples are first laid out on a long wide iron tray and placed in a drying room where they are thoroughly dried. After drying they are crushed to a granulated size—the ore so that it will pass through a 100-mesh

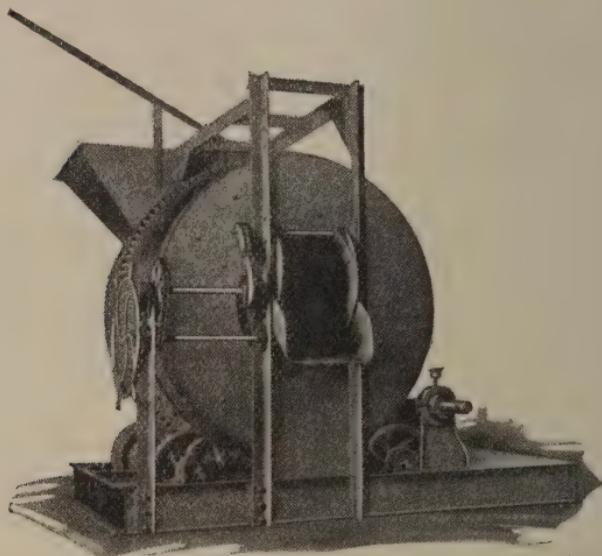
screen and the coal through an 80-mesh screen. The samples are then bottled and placed in a steel carrying case to be sent to the laboratory for analysis.

The tangential bins are so arranged that their discharge outlets are directly over a larry track. Each bin has six gate outlets, operated automatically by an arm on the larry car. The bins, being steel, freeze up very easily in cold weather and to prevent this as much as possible steam pipes are run along the outer surface. A scale hopper larry car runs along below the bins, taps the charges from the bins and carries the proper amounts of the various materials to the mixing stations and there discharges the contents of its bins.

Mixing of the ores and coal is accomplished by having three mixing stations, each of which has two Ransome mixers. These mixers are located at a level below the larry track. The larry drops the ores and coal to a bin and from there it is fed to the mixers. The mixers are revolving cylinders, with interior blades that toss and mix up the contents and finally carry it to the opposite end where it falls into a trough leading to a skip. This is the only process that the raw materials are subjected to before being used in the furnaces.

The mixed coal and ore coming from the mixer falls into the skip. One charge for the furnaces, known as the firing coal charge, is placed directly above the skip and may be tapped directly into the skip without going through the mixer. The skip is a rectangular steel car having the contents of one furnace charge. It runs by means of small iron wheels upon a railed incline, being propelled by means of an iron cable actuated by pulleys and electric motor. At each of the three mixing stations there are two mixers, two skips and

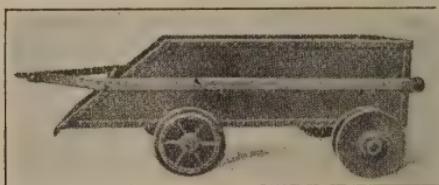
thus two runways up the incline to the top of the furnace building.



(Courtesy of Ransome Machinery Co.)

FIG. 21.—Mixer used for preparing the furnace charge.

The empty skip coming down the one track acts as a counterbalance to the loaded skip ascending on the other track. The skips are dumped at the top of the fur-



(Courtesy of Ransome Machinery Co.)

FIG. 22.—Skip car.

nace building by having the front wheels follow a descending track while the rear wheels are allowed to

remain on an outer and ascending track, lifting the rear of the skip to an inverted position. The contents are dumped into furnace bins near the top of the furnace building.

The furnace bins are large wood and steel structures, located at the top of the furnace building and in the center of the row of furnace blocks, enabling the discharge of material to both sides of the furnace building.

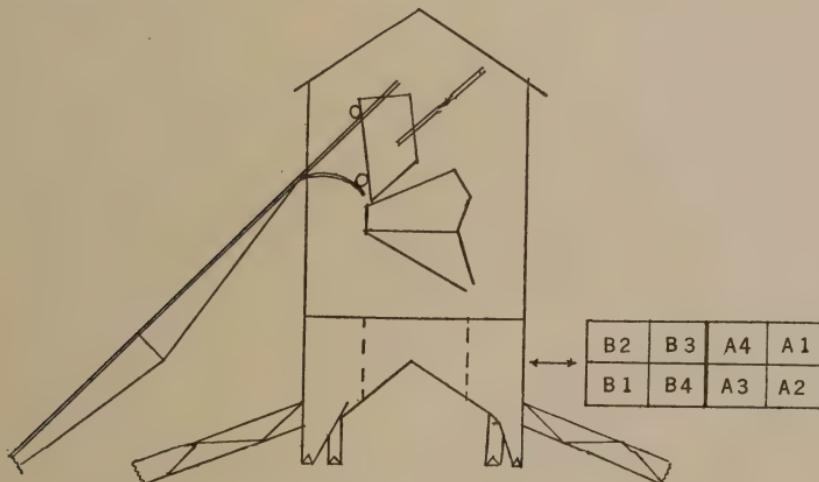


FIG. 23.—Furnace bins and skip.

There are four furnace bins for each skipway and thus eight bins for each mixing station. The bins have four compartments, three of which are used for the various ore charges and one for the coal charge. The material comes down from the skip over a distributing slideway which guides it to the proper bin. These bins are not intended to be storage places for materials and never carry any surplus mixtures or coals, but are being constantly emptied by the furnace charge cranes, and are refilled as soon as emptied.

The charge crane is a hopper crane running on an elevated track along the side of the furnace building and above the level of the furnace hoppers, which, in turn, are located directly above the furnace blocks. The charge crane acts as a transfer agent for the material from the furnace bins to the furnace hoppers, which

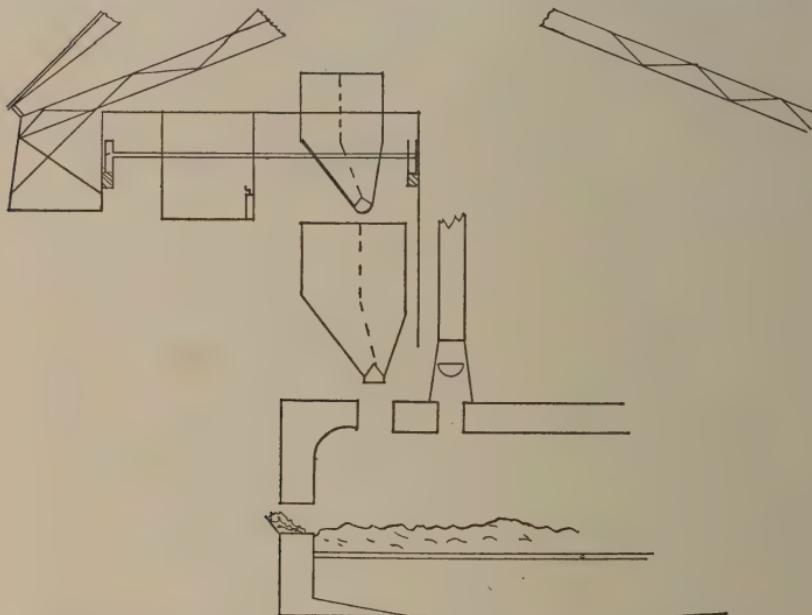


FIG. 24.—Charge crane. Located above the furnace hoppers.

in turn dump the charges of material directly into the furnaces. At each charging station directly below the furnace bins are four furnace bin gates for each side of the furnace, or an outlet for each furnace bin compartment. Three of the bin lead-outs are for ores and are extended further than the fourth which is for the coal charge. The gates are opened by means of an air-operated rod and a definite amount of material falls to the weighing hoppers of the charge crane. The material

falls into a partitioned hopper, one side for coal and the other for ore.

The charge crane deposits the ore and coal mixtures into the furnace hoppers, which in turn are arranged directly above the furnaces, one furnace hopper being arranged for each furnace door. The furnace hopper very much resembles the partitioned hopper of the charge crane as it has a partition through the middle, the inner compartment being for coal and the outer compartment for the ore charge. It tapers, as do all hoppers, to the bottom and at the termination of the neck it has a gate, which when swung in one direction opens the coal chamber and when swung oppositely it opens the ore charge section.

The furnace building of the plant is a steel structure extending about three-fourths of a mile, and is 35 feet wide. It houses thirty-eight furnace blocks. The furnace floor of the building is about 12 feet above ground level. This is done so that cars may be run under the furnace floor to receive and carry away the clinker or residues from the furnaces.

There are three types of the Eastern American Process style of furnaces used at this plant, namely the tunnel type, the four-furnace type and the six-furnace type. Structurally all of the furnaces are of the same measurements. Each furnace block has the same exterior measurements as all others. The differences in the types mentioned above come from the partitioning of the inside chamber of the furnaces.

The furnace blocks are "blocks" of firebrick, and masonry strengthened by steel rods and girders. The blocks are 45 feet long and 16 feet 6 inches wide and 7 feet high. The interior of the furnace contains a fur-

nace grate upon which the charge is placed and burned off, and an arched ceiling. In a four-furnace block, the interior of which is partitioned off into four separate and distinct furnaces, each furnace has hearth area of 19 feet $6\frac{1}{2}$ inches by 5 feet $11\frac{1}{2}$ inches. In the roof or ceiling of each furnace are three charge openings and two lead-off pipes 20 inches in diameter for the withdrawal of the zinc oxide laden gases. On the side or the face of the furnace are three working openings closed by doors. The hearth of the furnace is made of cast iron, either slotted or pin-hole grate bars. Below this is an ash pit constantly sealed with water to keep the hearth cool and through which is fed a blown draft of air.

In the operation of the furnace, supposing a charge having run for eight hours and being fully worked off, the stream of gases is cut off from the collection pipes by means of a butterfly valve just at the base of the flue. The ashes that were piled at the door or working opening to close the doorway are removed and thrown aside to be used again. The residuum remaining in the furnace after the charge has been worked out is in the form of luminous tough porous clinker. This red-hot clinker covering the whole grate is broken up by heavy hand slice bars and raked out of the furnace through the doors and dropped through an opening or doorway of the building floor, into cars below. The back walls of the furnace are then chiseled free from adhering clinker and the hearth is cleaned. The clean grate is quickly covered with coal dropped through the charging neck from the furnace hopper above. The doors of the furnace are closed and a small amount of draft is admitted through the ash pit. This is done

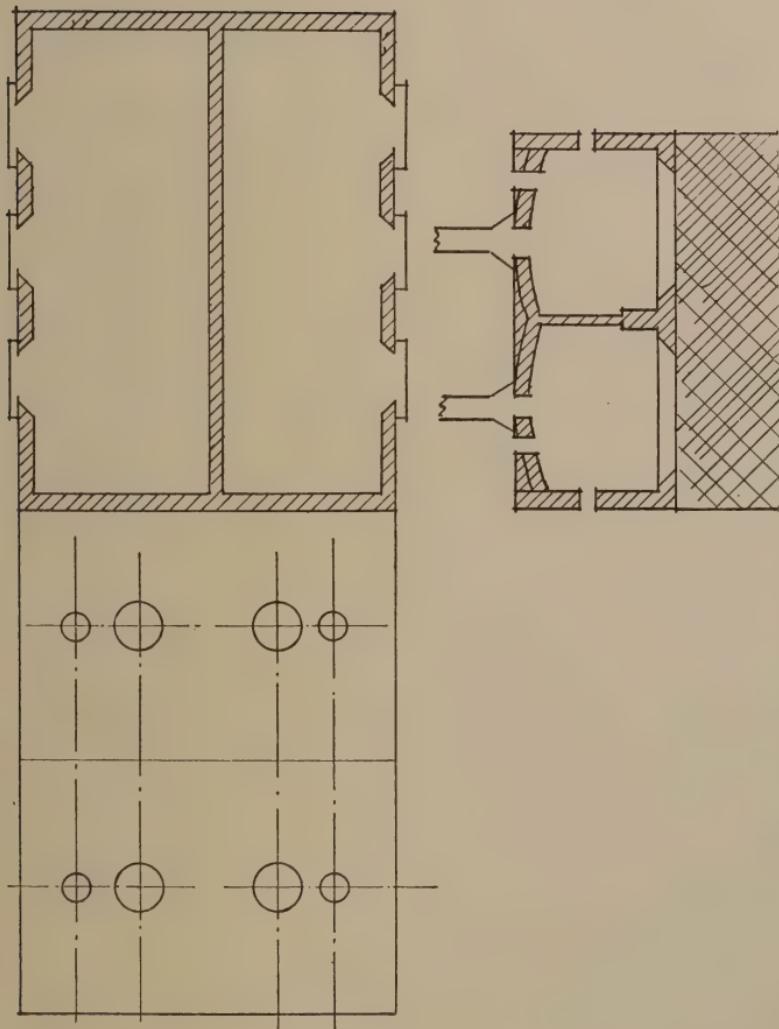


FIG. 25.—American Process zinc oxide furnace.

in order to heat up the coal bedding which will be used as the smelting medium for the next charge. When the bed of coal burns evenly and brightly the charge of ore and coal mixture, which is in the furnace hopper, is dropped into the furnace and spread out evenly over the whole grate area by means of long rakes. This burden usually runs 6 inches to 9 inches deep and will weigh about 55 pounds to 60 pounds per square foot. The temperature of the furnace at this point is about 700° C. The doors of the furnace are then closed by piling ashes on a suspended shelf of sheet iron, and the air gradually admitted under the grate until the whole charge is well ignited.

The draft or the 2-inch or 3-inch pressure of air that furnishes the cold blow to the ash pit is furnished by means of centrifugal electrical fans housed in a special fan house at one side of the furnace building. There is one fan for each furnace block. The draft is led from the fans through 12-inch sheet-iron tubing directly above the center of the block, where a main carries it down the length of the block. At each furnace doorway, or opening, branches of the air pipe come down the side of the furnace to the ash-pit door, where it is fed to the furnace, going up through the grates to aid in the combustion of the coal and to furnish oxygen for the formation of zinc oxide.

The charge within the furnace gradually rises in temperature, the gases of the combustion being led to the air because of the butterfly valve in the base of the flue, or discharging neck. After an hour or so the gases pouring from the furnace begin to show a yellowish color, tinged with green. The workmen call this flame "zinc candles." The early gases coming from the fur-

nace have a purplish color, being burning carbon monoxide gases. As the color of the flame grows to a deeper yellow it indicates that the reduction of the zinc and the formation of zinc oxide is progressing. After a while the flame becomes a deep yellow, tinged with green. This indicates a good oxide stream so the damper of the butterfly valve is closed and the stream of zinc oxide and gases allowed to be drawn upward through the collection pipes, called the down legs.

The charge burns for eight hours, the color of the gas stream passing upward through the damper indicating the stage of the fire and the grade of zinc oxide the furnace is producing. Sometimes the furnace will not produce good zinc oxide due to the fact that the charge may have been dropped before the fuel charge was sufficiently heated—thus a chilled fire, and consequently a poorer grade of zinc oxide. Again there may not be sufficient sulphur within the charge to sulphate the lead and cadmium and thus cause pinkish and yellowish discoloration of the pigment. The pressure of the cold blow draft is another influential factor upon the production and operation of the furnace, too much pressure may cause blow-holes in the charge allowing too rapid reduction in one location, overheating the grates and producing a variety of undesirable results.

Chemistry of the Formation of Zinc Oxide

The exact chemical reactions within the furnace at the time of the formation of zinc oxide are not known. The heat of the charge rises to 1100° C. to 1300° C. and it is practically impossible to have knowledge

of the actual reactions. It is presumed that the zinc content of the ore and the carbon of the coal unite to form CO and Zn. Continuing further the Zn and the CO form Zn and CO₂, the CO₂ combines with the Zn forming ZnO and CO. The CO gas thus formed acts as a very strong reducing agent upon the ore and increases the temperature of the furnace. The hydrogen of the water in the charge is also liberated by virtue of the oxygen content combining with Zn to form ZnO. The ores also contain a quantity of cadmium and lead and as these metals are volatilized they are also oxidized. Too much lead oxide, being yellowish and reddish in color, causes discoloration of the zinc oxide. The cadmium oxide is also distinctly yellow. However, these oxides of lead and cadmium are kept to a minimum by the action of the sulphur contained in the ore and in the coal. Sulphur, having a greater affinity for lead and cadmium, forms lead and cadmium sulphates, which are distinctly white. It is undoubtedly a fact that a portion of the zinc is also sulphated, but not to a great extent because chemical laboratory control of raw materials only permits enough sulphur to be used in the charge to afford proper care of the lead and cadmium. If any appreciable quantities of zinc sulphate form they are undoubtedly reduced to zinc oxide and to sulphurous anhydride, the latter completely escaping. Chemical changes are aided by the several agencies, such as the combustion of the coal mixed with the ore, the carbon monoxide formed in the furnace, the hydrogen liberated from the water of the mixture, and the exothermic reaction of the oxidation of the vaporized zinc. The oxidation of the zinc is supposed to take place near the ceiling of the furnace.

It was previously mentioned that there were three types of Eastern American Process zinc oxide furnaces used at this plant. Namely, the four-furnace type, the tunnel type and the six-furnace type.

The four-furnace type of block is characterized by the fact that it has four distinct furnaces within the block, the interior of the block being quartered. As all furnace blocks have six doors on each side there are three doors for each furnace, three charging necks and two gas necks. The grate area is 444 square feet for each

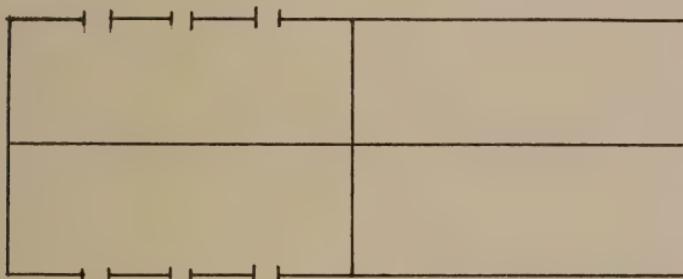


FIG. 26.—Four-furnace block division.

furnace. The advantage most cited for the four-furnace block is that it fits very nicely into an eight-hour shift schedule. It takes eight hours to burn out a charge so that by having a crew care for two blocks they are able to draw one furnace each hour. It also has an advantage in the fact that when a fire is drawn the cooling off of the furnace block is only one-fourth of the entire area of the block.

The tunnel type of furnace block is a type having the block divided by partitions into thirds. These partitions run from side to side forming three separate furnaces. This type of block has two doors for each side of the furnace, two charging necks per side and one

gas neck for each side, or two for the tunnel furnace. The grate area for this type of furnace is slightly greater than the four-furnace type as it does not have as many dividing walls. The grate area is 496 square feet per block. There are a few advantages of this type of block, one being that the absence of the back wall gives less trouble in drawing the worked-out charge, thus facilitating cleaning; another is the fact that structurally the tunnel type must have a higher ceiling arch, which does not reflect the heat so intensely upon

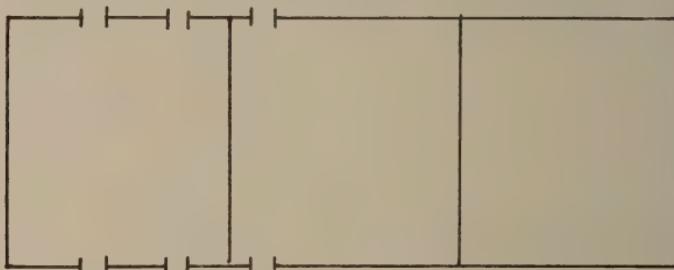


FIG. 27.—Tunnel furnace block division.

the charge and cause any fusible materials to "glass" the grates. There are several disadvantages, such as the severe cooling of the oxide stream because of shutting off one-third of the block at the time of charging. The larger arch also induces a consideration of higher maintenance costs.

The six-furnace type of block is one in which a center dividing wall has been run down through the funnel type of block. Thus there are six distinct furnaces made in the one block, each having two doors, two charging necks and one gas neck. The furnace, although not having such a large grate area as the other types, makes a greater ratio or recovery than the other

furnaces due to the fact that a constant temperature is usually maintained and that a constant and even mixture of the zinc oxide is always flowing into the main oxide stream. The six-furnace types are especially advantageous in the preparation of leaded zinc oxides where a high and even temperature is desired.

The zinc oxide stream drawn from the furnace through the gas neck, in combination with the burned gases, air, etc., goes directly upward through vertical sheet steel pipes, called down legs. The down legs are

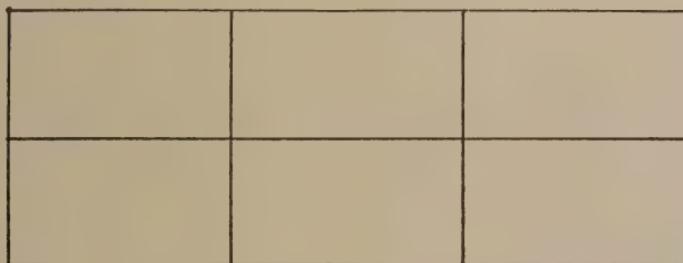


FIG. 28.—Six-furnace block division.

about 15 feet in height and at the upper end feed the zinc oxide into large cylindrical drums. The purpose of the drums is to thoroughly intermix the zinc oxide coming from the different furnaces of the block, and to permit the oxidation of any zinc vapors that escaped oxidation in the furnace, as well as to permit the gases to cool to some extent. There are either two or three drums which receive the zinc oxide first and these in turn feed the stream of gases and pigment to an upper and common drum.

By the time the oxide stream is drawn from the drums, which are directly above the furnace blocks, it is very well mixed. The temperature is about 1000° C., and the stream still contains some unoxidized zinc vapors.

In order to slowly cool this stream of gases and at the same time allow the zinc vapors to oxidize the gases are run through an extended pipe. The pipes are made of sheet steel, and are 4 feet in diameter. Each furnace block has considerable length of this pipe carrying the oxide stream and the intertwining arrangement of these large pipes, carrying the products of thirty-eight

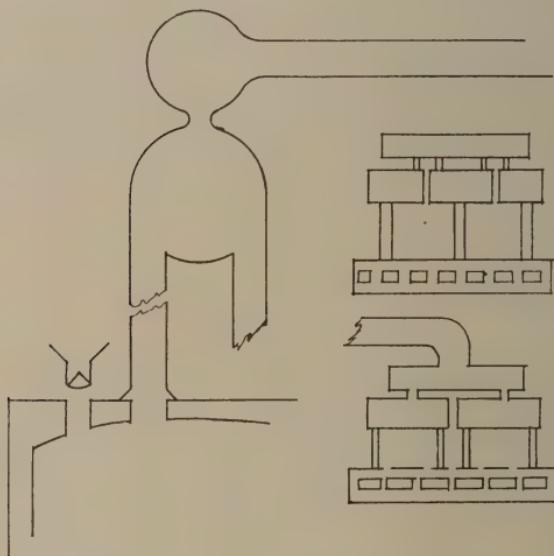


FIG. 29.—Down legs and drums.

blocks in a row, presents a rare spectacle. At certain intervals, along the length of the pipe, are little doors called ventilators. These ventilators permit the entrance of air which cools the gas stream and aids in the oxidation or any remaining zinc vapors.

Sediment forms in the pipes and at sections near the furnace building, where high temperatures are encountered, there may be semi-metallic deposits. This sediment and these deposits are very detrimental to the

main product, zinc oxide. Thus the pipes must be thoroughly cleaned periodically. This is done by cooling the furnace and sending men into the pipes to chisel loose and scrape away the accumulated sediment. This is done by picks, bar scrapers and special tools. The sediment, consisting of caked zinc oxide and

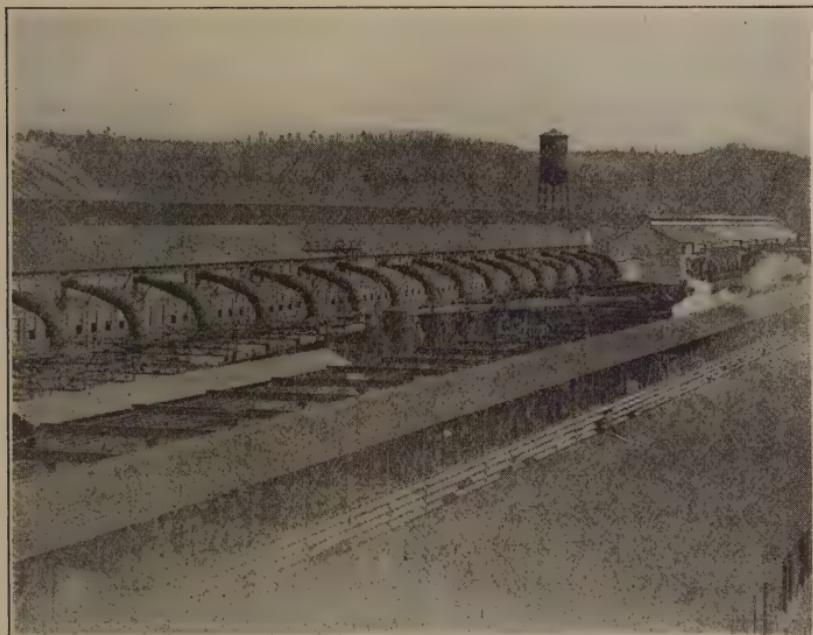


FIG. 30.—View of zinc oxide pipes carrying the zinc oxide laden gases from the furnace building.

metallic zinc, can be re-fed to the furnace and the zinc content reworked.

The drawing off of the oxide stream, as it forms in the furnace, and the propulsion of the gases and the pigment through the drums and the pipes is accomplished by means of a large fan. The large cooling pipes lead the zinc oxide streams to fans. The fans

are set in special fan houses located midway between the furnace buildings and the bag houses. The fans are of the centrifugal type, having closed gas inlets at both ends of the revolving axis. The fins of the fan blow the zinc oxide laden gases off through a tangent lead off pipe, through which it is led from the building. The fans are driven by electric motors and revolve at a speed of 350 to 400 r.p.m. They are mounted on a special water-cooled roller bearing as the temperature of the gases is so high that special care must be taken of the bearing surfaces. Each fan has several openings which permit periodical cleaning of any accumulations of zinc oxide, dirt, etc.

The zinc oxide stream is then led, by means of large sheet steel piping, to a cyclone blower. The cyclone blower is a mixing chamber where the dust particles of the oxide stream are blown in contact with vertical baffle plates, the forward motion temporarily arrested, and if too heavy for suspension in the moving gas stream, drop to the bottom of the cylindrical enclosure and from

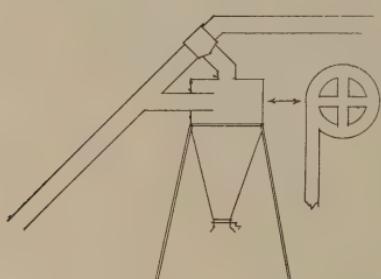


FIG. 31.—Cyclone blower.

there down to the conical base of the cyclone blower where it is immune from the blast of the moving gases above. The cyclone blower is suspended on a structural steel base, with legs of sufficient height to be on a level with the pipes carrying the oxide streams. The conical

collection section in the base is far enough above the ground level to permit a small railroad truck car to go beneath it for the removal of collected refuse.

The oxide stream is led off from the top center of the cyclone blower and is forced through a sheet-steel pipe to the bag house. There are two large bag houses at this plant the only difference being that at the one bag house the oxide stream is fed to the bottom of the bags and in the other it is fed at the top.

The main pipe leads the oxide stream into the top of the building and there feeds it to a large lateral pipe. This lateral pipe runs at right angles to the main incoming pipe and extends along the side of the building. It feeds ten sub-pipes, five on each side of the main incoming pipe. These sub-pipes carry the oxide stream across the width of the building. Each sub-pipe feeds nineteen circular openings to which are attached collection bags.

The bags are made of mesh muslin sheeting or tubing, so sewed as to become a tube 20 inches in diameter. The bags hang to a total length of 42 feet, being made of three sections of 14 feet each. They are made in sections so that if one part becomes defective it will not be necessary to replace the entire bag. The oxide laden gases enter the bag at the top and as they proceed downward the gases escape through the meshes of the bags but leave behind the solid particles of zinc oxide. The gases entering the bags are very hot, and if for some irregularity they are not sufficiently cooled they may cause the bags to ignite and result in a very destructive fire. As the gas stream comes into the building it is about 197° C., at the top of the bag it is about 168° C., half way down the bag the temperature has dropped to 77° C. and at the bottom of the bag the temperature will have dropped to 49° C.

The zinc oxide collects at the sides of the bags and

adheres to the cloth. As the bags get coated with zinc oxide there is a tendency to seal over the interstices of the meshes of the muslin and it is necessary to clean the bags of this accumulated zinc oxide so as to permit the escape of incoming gases. This cleaning is done by hand. By closing a damper each bag can be cut off from the incoming gases, and this also releases the pressure which usually holds the bags well extended. While



FIG. 31a.—View of zinc oxide plant. Showing pipes, cyclone and bag house.

they are cut off from the incoming gases, and hence hanging slack, a workman grabs hold of the side and by quick snappy shaking loosens the oxide which falls to the bottom hopper and into the collection bags at the floor level. The gas is then admitted to this bag and another one shut off and shaken. In this way the bags are kept clean on the inside and the pigment is collected into the small collection bags on the ground floor of the bag house.

When the collection bags at the bottom hoppers are sufficiently filled with zinc oxide they are removed,

tied shut by means of special colored strings, which mark the contents, and loaded on a small truck. Samples are taken so that by the time the pigment is ready to be packed it will have been classified and graded and will be used accordingly. A small electric truck makes up a train of the collection bag trucks and hauls them to the lower floor of the packing house. Here the bags are weighed, recorded and after labeling are allotted to the mixing room. After the destination of a lot is decided upon it is placed on an elevator and taken to the fourth floor of the building and placed at the side of the mixing hopper into which it is to go.

Each collection of zinc oxide at the bag house varies as to the time and the bag through which it is collected and this variation is sufficient to make some mixing necessary for the marketing of a standard product. The samples taken from the collection bags are taken to a testing room where they are rubbed down in oil, compared, graded for color and brightness and allotted for the mixtures. Thus the pigments are blended and a standard uniform product is procured.

From the wide shelf-like hoppers on the fourth floor the mixed zinc oxide falls into the bolting machines which are on the third floor. These are special zinc oxide bolting machines, fashioned after the flour bolting machines, having the zinc oxide led into the center of a revolving-screen cylinder, which is made of 16-20 mesh brass wire. The oxide is put through these screens not, as some people think, to regulate its fineness, but to pick out any pieces of foreign matter which might have gotten into it, such as pieces of bag, bag string, etc. As the pigment passes directly from the bolter to the packer it is essential that all foreign matter be removed at this point. Beside functioning as a

cleaning medium for the zinc oxide the bolter also acts as a mixing agent, mixing the zinc oxide to an even composition.

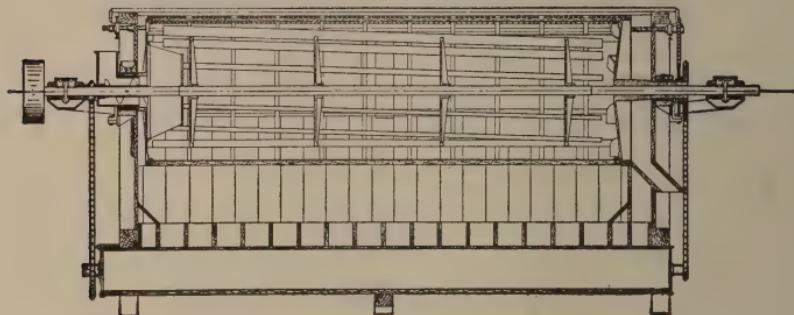


FIG. 32.

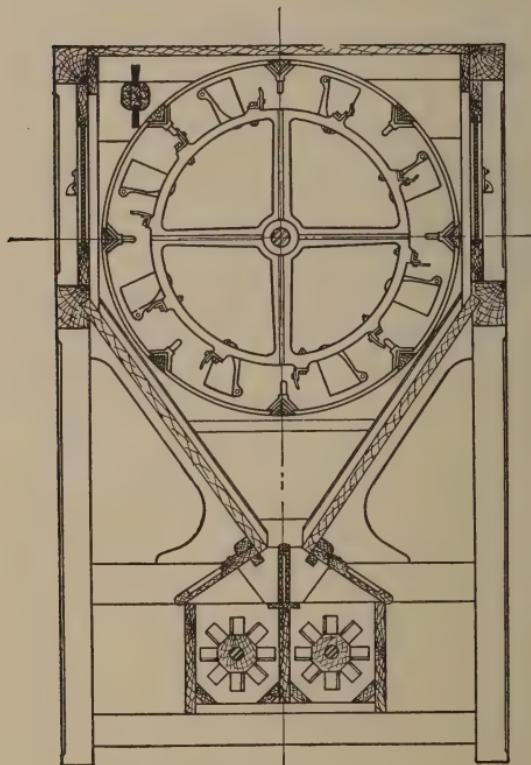
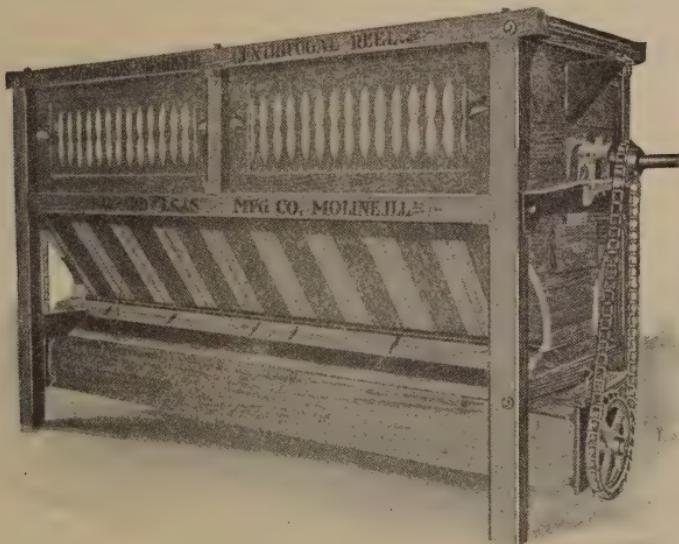


FIG. 32a.

FIG. 32 and 32a.—Cross and end sections of bolting machine.

The zinc oxide then drops to the hopper of the packing machine located on the floor below. The packing machine is the flour packing type, having a large feeding tube which leads the zinc oxide into a bag or barrel. Within this tube is an auger which rotates and forces the pigment down into the bag or barrel. The bag or barrel to be filled is placed on a counterweighted platform which is lifted so that the pigment feeds directly



(Courtesy of Barnard & Leas Mfg. Co.)

FIG. 32b.—Zinc oxide bolting machine.

to the bottom of the container. As the filling proceeds the platform is forced downwards and after it drops a given set distance it traps itself and throws the packing machine out of gear. The machine is adjusted so that it usually fills a given amount of pigment into a bag or barrel. The bags or barrels are removed from the packing machine platform and are weighed, more pigment added if necessary or some removed, so that the weight of the package is standard

at all times. The packages are tied, barrels headed and labeled on the packing floor, later to be trucked to a storage house from where they are shipped.

Zinc oxides made by the Eastern Type of American Process furnaces are principally the so-called lead free



(Courtesy of S. Howes Mfg. Co.)

FIG. 33.—Zinc oxide packing machine.

grades of the pigment. Low leaded and high leaded zinc oxides are also made. Lead-free zinc oxide is used in the rubber industry as a compounding pigment and in the paint industry as a pigment for use in conjunction with lead and other pigments.

Considerable time and money is being spent for researches into a means for producing American Process zinc oxide by better methods. The most notable development that so far has been made public was the work on a continuous furnace developed by means of the traveling grate. Briquetted coal is charged mechanically to a depth of 6 inches at the one end, it is ignited by the radiated heat of the furnace walls, and burned by an under-grate blast, and has an arrangement for carrying off the issuing gases. In this combustion chamber the temperature rises to about 800° C. to 850° C. As it travels along it forms the bed coal and underlies a briquetted ore charge fed continuously to a 3-inch depth. The charge, ignited by the bed coal, gives off gases which are carried off through a flue. In this second section the temperatures rises to about 1000° C. By the time the charge has been heated to a temperature for reducing the zinc contained in the ore charge the traveling grate has carried it to a separate chamber about 18 feet long. The under grate blast furnishes continuous air for the oxidation. The temperature of the main reducing chamber is about 1100° C. to 1250° C. The treated ore briquettes retaining their shape pass off at the tail end of the grate into a hopper where part of the fuel ash is screened off. The grate travels at a speed of 7 feet a minute. The use of ore-fuel briquettes has given hopes of reducing the amount of fuel necessary for a charge, and has given a greater percentage of recovery of the total zinc content.

A novel variation of the general design of the American Process furnaces and plants is found in a zinc oxide plant in Illinois. The raw material charge, composed of both the fuel and the ore, is raised to a storage bin

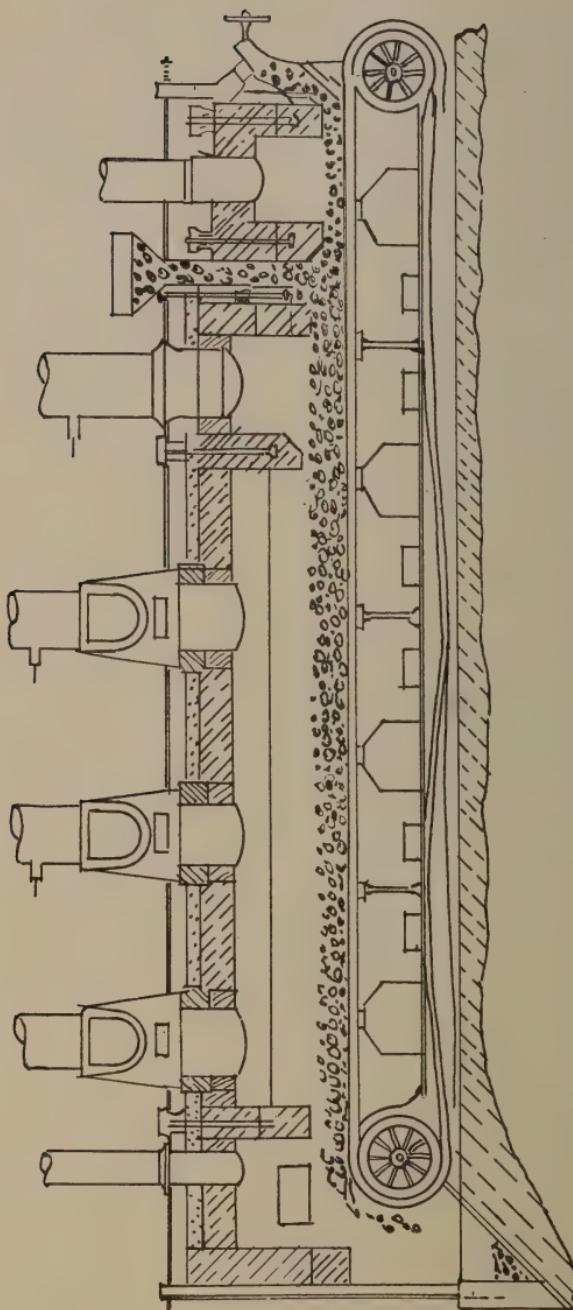


FIG. 34.—New traveling grate zinc oxide furnace.

by means of a bucket elevator. From this bin it is tapped directly into the buckets of a chain bucket trip conveyor. The conveyor takes the charging material out over the center of the block and by means of regulated trips dumps the contents into furnace bins which are below the conveyor and are directly above

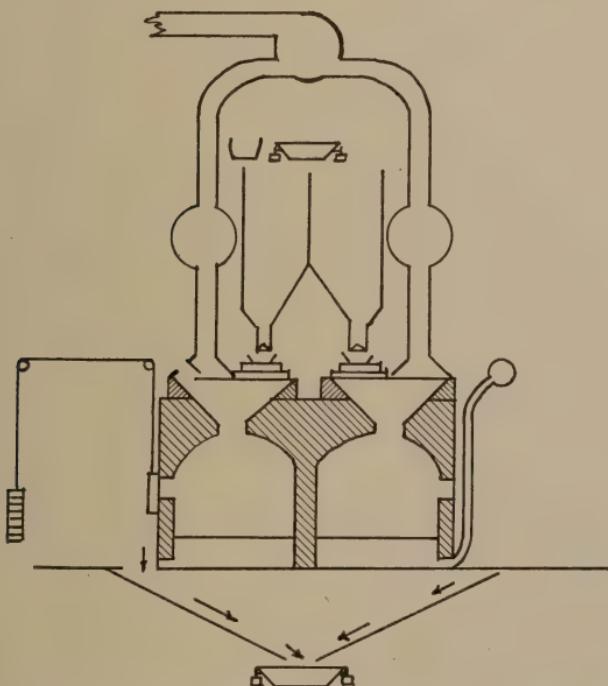


FIG. 35.—Novel American Process zinc oxide furnace.

the furnace block. This conveyor goes on down to the end of the building and returns beneath the furnace block carrying away the clinker from the collection hopper beneath the furnace floor.

The furnace used in this plant is of a four-furnace block type. It has the charge neck and the gas port combined in one furnace opening and thus only has the

one opening into the arch of the furnace roof. A counterbalanced raising door is used instead of the banked ash door. The clinker and ashes are removed by allowing them to fall through an open floor door, and into a bin, at the base of which runs the chain bucket conveyor.

The zinc oxide formed leaves the furnace through the charge opening, and goes to the down leg through a butterfly valve. The actual charge opening is covered over as soon as the charge has been dropped into the furnace. The down leg leads the zinc oxide into a side drum, there being a drum for each furnace of the block. From the side drums the gas stream is led to a top drum where the mixing of the entire stream of zinc oxide is effected.

The cooling pipes, with cooling ports every 10 feet, and fans are the same as previously described but the bag house and the system of collecting the zinc oxide differs in several respects.

The bag house is really an elevated building with a sheet metal hopper bottom. The stream of zinc oxide laden gases enter the side of the hopper and rises through a series of outlets which feed upward into bags. The bags are 10 inches in diameter and 15 feet long. They are mechanically shaken every 10 minutes by a cam and gear arrangement actuated by a revolving center rod. The shaking is timed by means of a cam gear arrangement on the fans. The shaking is effected by a solenoid causing the cams to come in contact with suspending arms of the bags and giving the bags an up and down jerky motion, and at the same time causing two metal rods which run along the side of the bag to slap up against it and thus

loosen all of the zinc oxide settled on the interior of the bag.

The zinc oxide falls into the large hopper below, which is V-shaped, and extends the width of the building. The zinc oxide is propelled along the base of the hopper

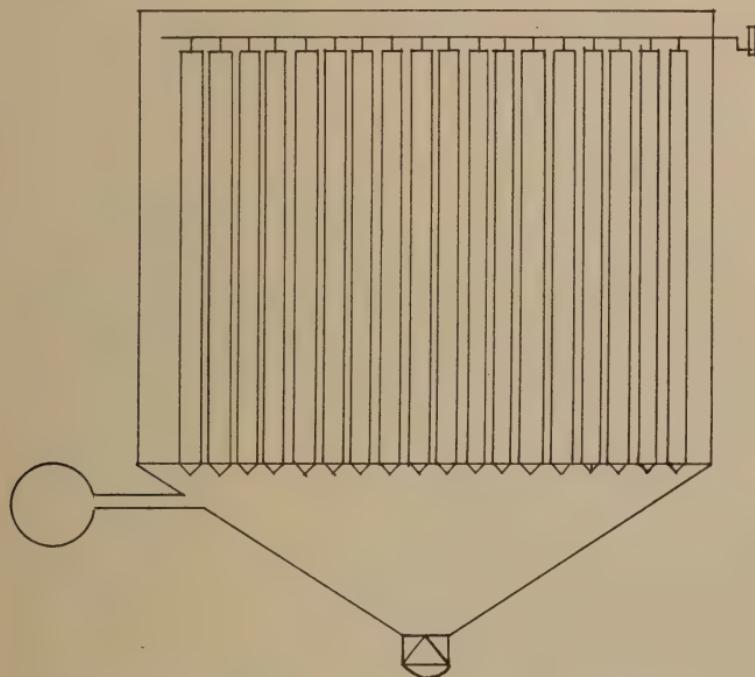


FIG. 36.—Novel bag house of an Illinois zinc oxide plant.

by means of a hand-operated screw propeller and at a central point it is tapped into small trucks. These collection trucks have an upright barrel-like body which may be covered at the top to keep the zinc oxide clean, and may easily be emptied by opening the gate at the bottom. The workmen very appropriately call the trucks "torpedoes."

CHAPTER 7

AMERICAN PROCESS ZINC OXIDE

Western Practice

The Western practice of producing zinc oxide by the American Process differs from the Easter practice principally in the furnace construction and operation.

The furnace block of a western type furnace is composed of usually eight to sixteen full width or "double" furnaces placed side by side, none of which have arches but are so constructed that all gases and fumes flow to a common arched roof. The furnaces have the air duct, water seal, ash pit, use the Wetherill pin-hole grates, and maintain a furnace temperature of about 1000° C. to 1100° C.

The operation of the furnace is by hand. The fuel charge is shoveled into the furnace from both sides, counterbalanced doors being used. When the fuel burns freely, being ignited by the residual heat from the previous charge, the ore charge of zinc ore and coal (usually half as much coal as ore) is shoveled into the furnace and spread to a thickness of 6 to 8 inches, and the cold blast slowly turned on. The furnace does not require any special attention for several hours, except that the charge be watched for blow-holes. After about six hours the charge is rabbled and in one hour later it is

rabbled again. The charge is reduced in eight hours and is then drawn.

The zinc fumes, the gases of combustion and the zinc oxide formed in the furnace rise to the arched roof of the block. All of the furnaces feed to the common arch, each furnace really being a partition of a large furnace block. From the furnace block the gases are led to the combustion chamber. The combustion chamber is a large firebrick chamber, the purpose of which is to thoroughly intermix all the zinc oxide coming from the furnace and to allow oxidation of any zinc vapor that remains. The combustion chamber, having an excess of free sulphur gases present, will cause lead oxide formed in the furnace to be converted into basic lead sulphate, which is white and accordingly does not have a tendency to discolor the pigment as would lead oxide. It also acts as a settling agent and any heavy particles are settled out in this chamber.

The cooling of the zinc oxide stream is effected by large pipes, the same as in the Eastern practice, and the zinc oxide collected in bags of the same design.

The Western furnaces use principally roasted blende and oxide ores, and as fuel they use a semi-bituminous coal, although several plants now in operation use coke breeze and anthracite coal as fuel. The principal products of the Western furnaces are leaded zinc oxides. Temperatures are relatively constant and by uniform procedure a high quality uniform leaded zinc oxide can be produced.

One of the most recently constructed Western practice American Process plants is at Murray, Utah, and is owned by the Utah Zinc Company.

The zinc ore used at this plant is a low lead content,

20 per cent zinc ore, mined in Utah. The ore is brought to the plant by rail and dumped from the cars on a trestle and allowed to weather without rehandling. It is later placed on a 20-inch belt conveyor which carries it to the crushing plant.

The crushing plant is designed to handle 150 tons of ore each eight-hour shift. The ore is passed over a $\frac{3}{4}$ -inch grizzly and thence to a 10-inch by 20-inch Allis Chal-

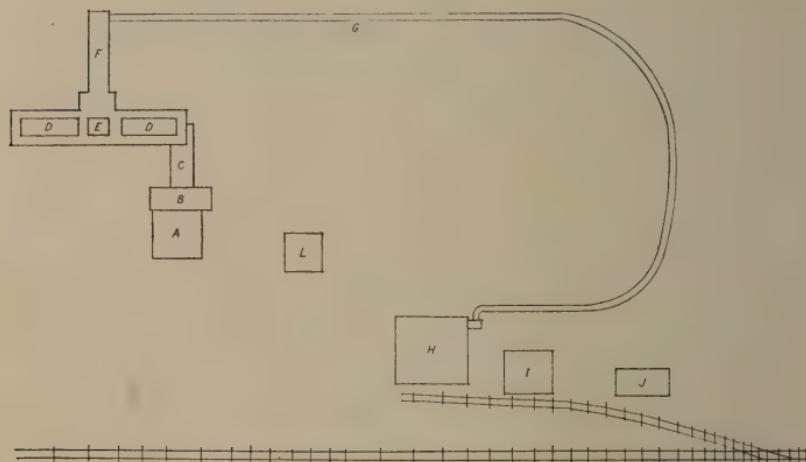


FIG. 37.—Zinc plant at Murray, Utah.

mers Blake crusher. The crushed ore is elevated by means of a bucket elevator to a $\frac{1}{4}$ inch square mesh trommel, the oversize from which falls into a set of 14-inch by 30-inch rolls and from the rolls to the foot of the elevator. The crushed ore that passes the trommel goes directly to a large crushed ore storage bin capable of holding about 350 tons of crushed ores. The different ores going into the storage bin are distributed by means of an 18-inch belt conveyor which in turn feeds it to shuttle conveyors.

The ore is drawn from the storage bin into small trucks, weighed and dumped into a large concrete mixer. Coke breeze is also drawn from a storage bin, weighed and dumped into the mixer. Water sufficient to give proper moisture content is also placed in the mixer and the charge is given five minutes' churning.

The mixed ore and coke is elevated by means of a second 60-foot bucket elevator to the mixed ore bin. The bin is designed to store 150 tons of mixed ore. A portion of this bin is also used for coke storage—the fuel charge of the furnace.

The fuel charge of coke and the burden charge of mixed ore and coke are drawn from the storage bin into the charge cars that are low and wide, and which are so designed with hinged lip that the side can be turned down to form a shelf flush with the bottom. These cars are run up alongside the furnace and the contents shoveled into the furnace doors in charging the individual sections.

The furnace building is constructed of concrete and structural steel, having its roof and sides made of corrugated iron. The foundations are all concrete and support the furnace and operating floor, which is also concrete and is elevated 7 feet 3 inches above the ground level.

The furnace consists of two volatilizing sections flanking a central section, which is the combustion chamber. The furnace is so designed that either section can be cut off from the combustion chamber if it is so desired. There are sixteen hearths in the furnace, eight in each section. The concrete foundation beneath each section forms individual chambers 9 feet 2 inches deep and 6 feet 6 inches by 13 feet in area. These enclosed

chambers are filled with water and serve the purpose of cooling the grates, and thus preventing buckling, and for collecting any ashes that may pass through the Wetherill grates. The water for the chambers is supplied through a $\frac{3}{4}$ -inch feed pipe controlled by a valve operated from the charging floor. An overflow is provided immediately below the blast inlet and 14 inches below the grates. A water-tight door near the bottom of the chamber can be removed to clean out the accumulation of ashes which in time may become troublesome.

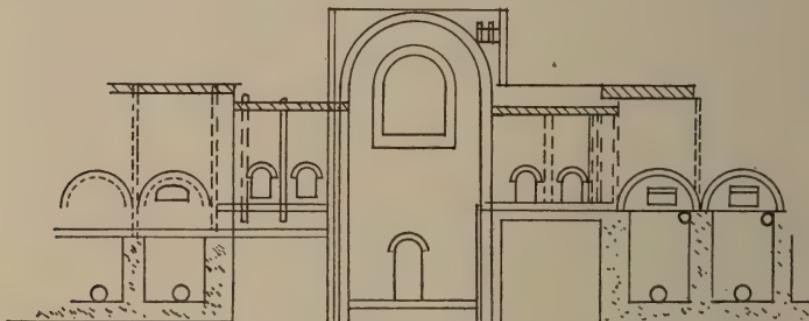


FIG. 38.—Western type of zinc oxide furnace.

The grates of the furnace rest on I beams supported by the concrete. An 8-inch blast pipe enters each chamber just below the grate level. The furnace is constructed of common brick, lined on the inside by firebrick. The top of the main arch is 8 feet 6 inches above the top of the grates while the smaller arches are 40 inches high and extend $3\frac{1}{2}$ feet into the furnace. The furnace is substantially built and is reinforced by brick, stays of channel and I beams and $\frac{7}{8}$ -inch rods. A firing or feeding door is provided for each of the short arches, making sixteen doors for each section of the furnace.

The earlier designs of the Western type furnaces had doors along one side of the furnace only, rather than the two rows of doors opposite each other. The later design of doors on both sides of the furnace has a disadvantage in that it permits the furnace to cool rapidly during cleaning and charging periods, but this is possibly offset by the facility with which accretions can be removed and the ease with which recharging can be done. The furnace not having a back wall, permits the side walls to be cleaned easily and allows for more rapid cleaning of the grates.

The grates are of the Wetherill type, similar to those used in all American Process zinc oxide furnaces. They are cast iron with tapering perforations $\frac{1}{8}$ inch in diameter at the top surface and are spaced 1 inch in both directions. The combustion chamber is a large brick chamber connected on either side by brick passages, or "bridges" to the furnace sections where the zinc oxide is volatilized. In the sides of these passages, or bridges, are small arched openings, like windows. These windows are checkered with brick masonry and removable bricks are provided to allow the regulation and flow of fresh air into the combustion chamber.

Once the furnace is in operation the procedure consists of drawing and recharging in rotation the individual doors of the furnace hearths. The procedure is as follows: The blast is cut off from the particular furnace section to be cleaned and the furnace is opened. The loose part of the last charge is raked off by hand and deposited on the floor to be used for recharging later. The semi-fused clinker is removed in a fashion similar to the cleaning of fires under a coal-fired boiler, a slice bar being used to break it up and a rake used to

draw it from the furnace. The clinker is dropped through a hole in the floor directly in front of the furnace door, to a car beneath in which it is transported to a dump. A layer of freshly moistened coke breeze is shoveled into the furnace and spread to an even depth on the cleaned grate. The blast gate is partly opened to admit air. The residual heat from the hot brickwork of the arch ignites the fuel and when it is burning briskly the material saved from the prior furnace charge, with a quantity of ore and coke mixture from the recharge car is shoveled into the furnace and spread evenly over the bed of fuel. The furnace door is then closed and the full blast turned on. This completes the charging and the furnace men then proceed to repeat the operation in the next furnace division, and so continue around the furnace working the sixteen divisions in succession. In the presence of carbon in the form of coke the zinc of the ore is first reduced and volatilized; it is then oxidized by coming in contact with an excess of air in the large arch and in the combustion chamber. A brick down-take conveys the fume-laden gas to a rectangular cooling flue which is also made of brick.

The air pressure in the blast beneath the furnace grates is equivalent to 4 inches of water. This blast is supplied by a Sturtevant multivane fan of a capacity of 40,000 cubic feet per minute at a speed of 460 r.p.m.

From the combustion chamber down-take the zinc oxide laden gas is drawn into a cooling flue. The cooling flue is a circular sheet iron pipe 45 inches in diameter and 1000 feet long. It is supported by low bouts about 5 feet above the ground. A shorter flue is undesirable because it does not give the gases suf-

ficient time to cool. Elbows and sharp turns are avoided because they offer unnecessary resistance to the flow of gas. Expansion joints are provided every 30 feet while at intervals of 8 feet along the bottom of the pipe are small openings fitted with sliding gates. Through these gates any oxide that settles may be withdrawn when necessary. The draft, however, is such that this accumulation does not amount to much.

The flow of zinc-laden gas through the cooling flue is effected by a 140-inch Sturtevant exhaust fan situ-

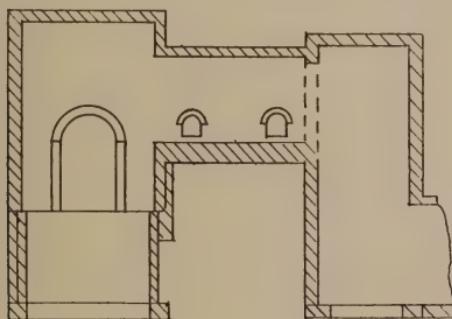


FIG. 39.—Combustion chamber and down-take.

ated at the end of the flue near the bag house. The fan has a capacity of 70,000 cubic feet per minute at 300 r.p.m. and creates $1\frac{1}{3}$ ounces per square foot in effluent gas. The high temperature of the gases make it necessary to have the bearings on the fan water cooled.

The bag house, in which the zinc oxide is collected, is 100 feet by 70 feet and has a wall 22 feet high. The zinc-laden gases enter at the bottom of the building and are distributed through the main pipes and laterals to 528 seamless cotton bags. The bags are 22 inches in diameter and are 28 feet high. The lower and open

ends of the bags fit snugly around the collars on the distributing pipes while the upper ends of the bags are closed and are supported by short ropes or small chains suspended from the grillwork below the trusses of the roof. The meshes of the bags collect the zinc oxide and allow the gases to escape.

The zinc oxide is shaken loose from the bags and is collected in bags attached to the under surface of the distributing pipes. When these collection bags are full they are detached and the contents hauled to a 9-inch extra heavy screw conveyor which carries the oxide horizontally 70 feet to the building where the packing is done. Here it is elevated by means of an 8-inch bucket elevator to a small storage bin from which it is drawn for packing.

CHAPTER 8

SPECIFICATIONS FOR THE PHYSICAL AND CHEMICAL QUALITIES OF ZINC OXIDES

STANDARD METHOD OF SAMPLING ZINC OXIDE

Product: U. S. Pharmaceutical Zinc Oxide.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Brightness.....	2
Smoothness and freedom from specks..	3
Hiding power.....	4
Settling in water.....	5

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by the standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in HCl.....	0— .010	4
H ₂ O at 110° C.....	0— .150	3
SO ₃ (total S as).....	0— .060	5
H ₂ O soluble salts.....	0— .200	2
Cl.....	0— .020	1
PbSO ₄ (total Pb as)....	0— .040	9
As ₂ O ₃	0— .005	39
ZnO.....	99.0—100.0	10
H ₂ O sol. alkali.....		11
Total heavy metals.....		12

Product: French Process Zinc Oxide.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Brightness.....	2
Smoothness and freedom from specks..	3
Hiding power.....	4
Settling in water.....	5

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by the standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in HCl.....	0.0.03	4
H ₂ O at 110° C.....	0-0.25	3
SO ₃ (total S as).....	0-0.25	5
H ₂ O soluble salts.....	0-0.75	2
Cl.....	0-0.06	1
PbSO ₄ (total Pb as).....	0-0.55	7 or 8

Product: Lead Free Zinc Oxide for Rubber Compounding.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Smoothness and freedom from specks..	3
Hiding power.....	8

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by the standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in acetic acid.....	0-0.25	16
Insoluble in HCl.....	0-0.100	4
H ₂ O at 100° C.....	0-0.40	3
SO ₃ (total S as).....	0-1.00	5
SO ₂ (reducing power equiv. to)	0.20	13
H ₂ O soluble salts.....	0-1.50	2
Cl.....	0-0.10	1
PbSO ₄ (total Pb as).....	0-0.40	7 or 8

Product: Superfine Rubber Makers, Zinc Oxide.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Particle size.....	15
Color.....	11
Tensile strength and elongation.....	12
Abrasion resistance.....	13
Rate of cure.....	14

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by standard method of analysis specified.

	Limits Per cent	Analysis Number
H ₂ O at 110° C.....	0.00–0.75	3
Insoluble in acetic acid..	0.00–0.15	14
PbO (total Pb as).....	0.10–0.25	8

Product: Lead Free Zinc Oxide for Paint Manufacturing.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Brightness.....	2
Smoothness and freedom from specks..	3
Hiding power.....	4
Tail length.....	6

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in HCl.....	0-0.040	4
H ₂ O at 110° C.....	0-0.40	3
SO ₃ (total S as).....	0-0.45	5
SO ₂ (red power equiv. to)..	0-0.20	13
H ₂ O sol. salts.....	0-1.25	2
Cl.....	0-0.10	1
PbSO ₄ (total Pb as).....	0-0.82	7 or 8

Product: Five Per Cent (5%) Leaded Zinc Oxide.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Brightness.....	2
Smoothness and freedom from specks..	3
Hiding power.....	4
Tail length.....	6

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in HCl.....	0-0.15	4
H ₂ O at 110° C.....	0-0.40	3
Total SO ₃ (min.).....	0-0.70	5
SO ₂ (red. power equiv. to)	0-0.30	13
H ₂ O soluble salts.....	0-1.25	2
PbSO ₄ (total Pb as).....	3.0-5.5	6 or 7

Product: Thirty-five Per Cent (35%) Leaded Zinc Oxide.

The physical properties of this product must conform to standard samples when compared by the methods specified.

	Physical Test Number
Color.....	1
Brightness.....	2
Smoothness and freedom from specks..	3
Hiding power.....	4
Tail length.....	6

The Chemical Properties.—The chemical constituents of this product, as listed below, must be within the limits indicated when tested by standard method of analysis specified.

	Limits Per cent	Analysis Number
Insoluble in HCl.....	0- 0.30	4
H ₂ O at 110° C.....	0- 0.40	3
Total SO ₃ (min.).....	5.50	15
SO ₂ (red. power equiv. to)	0-0.70	13
H ₂ O sol. salts.....	0-1.20	2
PbSO ₄ (total Pb as).....	33.0-37.0	6 or 7

STANDARD METHOD OF SAMPLING ZINC OXIDE

Shipping, receiving or arbitration samples from packages that have been closed shall be taken with a long, narrow sampling scoop, approximately 25 inches long, the section being an arc approximately 1 inch across and $\frac{3}{4}$ inch deep. This scoop shall be inserted at the top of the barrel at a point not more than 6 inches from the chime, or through a stave within 12 inches of the one end, and thrust obliquely through the contents of the barrel. The sample thus cut out shall be of length equal to at least half the height of the package.

In taking samples from bags, the scoop shall be inserted near the edge of the surface of the contents, and thrust obliquely through the material. The samples thus cut shall be of length equal to at least two-thirds the height of the package.

If a moisture determination is to be made, at least half a pound sample shall be taken, as above described, and immediately placed in an air-tight receptacle, which it must fill.

CHAPTER 9

STANDARD METHOD OF TESTING THE PHYSICAL QUALITIES OF ZINC OXIDE

PHYSICAL TESTING METHOD NUMBER ONE FOR THE PROPERTY OF:—COLOR.

Description:

Approximately 5 grams of the sample shall be thoroughly mixed with the smallest quantity of bleached linseed oil that will produce a smooth paste. This paste shall be spread on a palette of colorless plate-glass in a smooth and even layer, that will not transmit light and is at least 1 inch by 3 inches in area.

An equal amount of the standard sample shall be prepared in the same way, care being taken to bring the paste to the same consistency as the sample being tested. This paste shall be spread in a similar manner on the palette beside the sample, touching it, and the two compared in diffused daylight. In doing so, the palette shall be tilted so that the light will strike the surface of the pastes at different angles and the under surfaces shall also be observed through the glass.

In doubtful cases only the sample and one standard sample may be spread on the palette at the same time, and their edges must touch.

To be on-grade the sample must be as white as the standard.

PHYSICAL TESTING METHOD NUMBER Two
FOR THE PROPERTY OF:—BRIGHTNESS.

Description:

Five grams of the sample and 1.20 grams of bleached linseed oil shall be mixed to a smooth, uniform paste. The oil used may be determined by dropping from a point which has been standardized by counting the number of drops necessary to weigh 1.20 grams. The dropping shall be at a rate not greater than 70 drops per minute. All the sample and all the oil must be thoroughly incorporated. This paste shall be spread on a palette of colorless plate-glass in a smooth and even layer that will not transmit light and is at least 1 inch by 3 inches in area.

When the nature of the pigment requires more oil than above noted, more shall be used but comparisons shall be made only between samples which have a like oil-vehicle ratio.

An equal amount of the standard shall be prepared in the same way. This paste shall be spread in a similar manner on the palette beside the sample, touching it, and the two compared by observing in diffused daylight the two samples by looking through the glass.

In doubtful cases only the sample and one standard may be spread on the palette at the same time, and their edges must touch.

To be on-grade the sample must be equal to or better than the standard in brightness or brilliancy.

PHYSICAL TESTING METHOD NUMBER THREE
FOR THE PROPERTY OF:—SMOOTHNESS AND
FREEDOM FROM SPECKS.

Description:

Approximately 5 grams of the sample shall be thoroughly mixed with the smallest quantity of bleached linseed oil that will produce a smooth paste. This paste shall be spread on a palette of colorless plate-glass in a smooth and even layer that will not transmit light and is at least 1 inch by 3 inches in area.

An equal amount of the standard shall be prepared in the same way, care being taken to bring the paste to the same consistency as the sample being tested. This paste shall be spread in a similar manner on the palette beside the sample, touching it, and the two compared in diffused daylight. In doing so, the palette shall be tilted so that the light will strike the surface of the pastes at different angles.

In doubtful cases, only the sample and the one standard may be spread on the palette at the same time, and their edges must touch.

To be on-grade the sample must contain no more granular or foreign matter than the standard. This is to be determined by the feel under the knife and the amount of noise made in the rubbing down and by observation of the surfaces after the pastes have been spread on the palette.

PHYSICAL TESTING METHOD NUMBER FOUR
FOR THE PROPERTY OF:—HIDING POWER.

Description:

Five grams of the sample, 0.5 gram of ultramarine blue and 1.20 grams of bleached linseed oil shall be mixed to a smooth uniform paste of uniform color throughout. The oil used may be determined by dropping from a point which has been standardized by counting the number of drops necessary to weigh 1.20 grams. The dropping shall be at a rate not greater than 70 drops per minute. The mixing shall be done by rubbing lightly with a spatula the blade of which is not over 5 inches long. All the sample, blue and oil must be thoroughly incorporated. This paste shall be spread on a palette of colorless plate-glass in a layer that will not transmit light.

The standard shall be prepared in the same way and spread in a similar manner on the palette beside the sample, touching it.

Another standard paste shall be prepared by mixing 5.5 grams of the standard, 0.5 gram of ultramarine blue and 1.32 grams of bleached linseed oil in the same way and spread in a similar manner on the palette beside the sample, touching it.

In doubtful cases, only the sample and the two standards may be spread on the palette at the same time and the sample must be in contact with both standards at the edges.

To be on-grade the sample must not be darker than the first standard and not lighter than the second standard when observed through the glass.

PHYSICAL TESTING METHOD NUMBER FIVE
FOR THE PROPERTY OF:—SETTLING IN WATER.

Description:

This test shall be made in a flat-bottomed glass tube of $\frac{11}{16}$ inch diameter, 6 inches in height and uniform bore. Twenty-seven c.c. of water should fill it to a height of $4\frac{3}{4}$ inches with an allowance plus and minus variation of $\frac{1}{16}$ inch.

Five grams of the sample shall be put into the tube after the latter is half filled with water. The product and water shall be well mixed with a piece of wire about 14 or 12 gauge and the tube filled with water to a height of $4\frac{3}{4}$ inches. The wire is to be removed and washed off in the process of adding this water. The tube shall then be shaken vigorously eighty times, the thumb being held over the opening at the top. After shaking, it shall be placed in a rack so that it stands vertically and the height of the column of the product measured at the end of 1, 2, 3 and 24 hours.

An equal amount of the standard shall be treated in the same way.

To be on-grade the height of the column of the sample must be within 10 per cent plus or minus of the height of the column of the standard at the end of 24 hours.

PHYSICAL TESTING METHOD NUMBER SIX
FOR THE PROPERTY OF:—TAIL-LENGTH.

Description:

A sample of 400 grams is mixed and ground in mills with 120 c.c. of refined linseed oil having an acid number of not less than 5 nor more than 7. This mix is by weight 78 per cent pigment and 22 per cent oil. After standing an hour or so, 40 grams of the paste shall be weighed out and thinned down to a mixture of 40 per cent pigment and 60 per cent oil.

This mixture shall be stirred until the pigment is thoroughly incorporated in the oil to a uniform paint. Immediately after discontinuing the stirring a portion of the paint shall be drawn off into a glass pipette, having a $\frac{1}{16}$ -inch orifice. The tip of the pipette shall be wiped off and the paint column is to stand in it at a height of 2 inches above the tip. Then holding the pipette vertically $\frac{1}{2}$ inch above the clean, dry surface of a glass plate, 4 drops are allowed to fall on the plate. The latter shall immediately be placed in a vertical position and the paint allowed to run down it for exactly two minutes.

A similar sample of the standard shall be treated in exactly the same way.

To be on-grade the over-all tail-length of the sample must be within 20 per cent, plus or minus, of the over-all tail-length of the standard.

PHYSICAL TESTING METHOD NUMBER EIGHT
FOR THE PROPERTY OF:—HIDING POWER.

Description:

Five grams of the sample, 0.5 gram of ultramarine blue and 1.20 grams of bleached linseed oil shall be mixed to a smooth uniform paste of uniform color throughout. The oil used may be determined by dropping from a point which has been standardized by counting the number of drops necessary to weigh 1.20 grams. The dropping shall be at a rate not greater than 70 drops per minute. The mixing shall be done by rubbing lightly with a spatula the blade of which is not over 5 inches long. All the sample, blue and oil must be thoroughly incorporated. This paste shall be spread on a palette of colorless plate-glass in a layer that will not transmit light.

The standard shall be prepared in the same way and spread in a similar manner on the palette beside the sample, touching it.

Another standard paste shall be prepared by mixing 5.75 grams of the standard, 0.5 gram of ultramarine blue and 1.38 grams of bleached linseed oil in the same way and spread in a similar manner on the palette beside the sample, touching it.

In doubtful cases, only the sample and the two standards may be spread on the palette at the same time and the sample must be in contact with both standards at the edges.

To be on-grade the sample must not be darker than the first standard and not lighter than the second standard when observed through the glass.

PHYSICAL TESTING METHOD NUMBER NINE
FOR THE PROPERTY OF:—OIL ABSORPTION.

Description:

The sample to be tested shall first be placed in a small wide-mouthed bottle and well shaken so as to eliminate any packed particles or lumps.

Twenty grams of the pigment shall be weighed into a flat-bottomed glass. Refined linseed oil shall be added drop by drop from a burette. As the oil comes in contact with the pigment, the dry pigment which has not been wet should be lifted from the outer edge and placed over the oil so as to bring all the oil surface in contact with the pigment. This should be accomplished by lifting the pigment with a small spatula and lightly dumping it over the oil, at the same time giving the glass a circular motion and avoiding any pressure on the pigment. When the pigment particles become wet with oil, they tend to coalesce and form small lumps of paste. As the absorption of oil progresses these lumps of paste by taking up more pigment and matting together form larger lumps which when stirred around form balls. When this point is reached the rate and quantity of the oil addition shall be decreased until only a few drops of oil are added at a time. In adding oil at this point it should be allowed to strike on the lumps and not on the remaining dry pigment. After each oil addition these lumps are lightly stirred so as to bring the oil surface in contact with the remaining dry pigment. With the further addition of oil and further stirring, these balls will join together and form one large lump with but little dry pigment remaining. This point is close to the end point and the oil should be

added very carefully, only one or two drops at a time. When the last of the dry pigment has been picked up and wet the end-point is reached.

An equal amount of the standard shall be tested in the same manner, and under the same conditions, especially temperature, care being taken to manipulate the test in the same manner, and to obtain a ball of the same consistency.

The number of cubic centimeters of oil required to reach the end-point is considered the oil absorption of the pigment.

To be on-grade the sample must lie within a range the maximum of which is .30 c.c. more than the oil absorption of the standard sample and the minimum of which is .20 c.c. less than the standard sample.

PHYSICAL TESTING METHOD NUMBER TEN
FOR THE PROPERTY OF:—OIL ABSORPTION.

Description:

The sample to be tested shall first be placed in a small wide-mouthed bottle and well shaken so as to eliminate any packed particles or lumps.

Twenty grams of the pigment shall be weighed into a flat-bottomed glass. Refined linseed oil shall be added drop by drop from a burette. As the oil comes in contact with the pigment, the dry pigment which has not been wet should be lifted from the outer edge and placed over the oil so as to bring all of the oil surface in contact with the pigment. This should be accomplished by lifting the pigment with a small spatula and lightly dumping it over the oil, at the same time giving the glass a circular motion and avoiding any pressure on the pigment. When the pigment particles become wet with oil, they tend to coalesce and form small lumps of paste. As the absorption of the oil progresses these lumps of paste by taking up more pigment and matting together form larger lumps which when stirred around form balls. When this point is reached the rate and quantity of the oil addition shall be decreased until only a few drops of oil are added at a time. In adding oil at this point it should be allowed to strike on the lumps and not on the remaining dry pigment. After each addition of oil these lumps are lightly stirred so as to bring the oil surface in contact with the remaining dry pigment. With the further addition of oil and further stirring, these balls will join together and form one large lump with but little dry pigment remaining. This point is close to the end-

point and the oil should be added very carefully, only one or two drops at a time. When the last of the dry pigments has been picked up and wet the end-point is reached.

An equal amount of the standard shall be tested in the same manner, and under the same conditions, especially temperature, care being taken to manipulate the test in the same manner and to obtain a ball of the same consistency.

The number of cubic centimeters of oil required to reach the end-point is considered the oil absorption of the pigment.

To be on-grade the sample must lie within a range the maximum of which is .60 c.c. more than the oil absorption of the standard sample and the minimum of which is .10 c.c. more than the standard sample.

PHYSICAL TESTING METHOD NUMBER ELEVEN
FOR THE PROPERTY OF:—COLOR IN RUBBER.

Description:

The sample shall be incorporated in a rubber compound of the following composition by weight:

First latex pale crepe.....	920
Sulphur.....	55
Hexamethylenetetramine.....	6
Zinc oxide.....	1260

The compound shall be cured in mold for sixty minutes at 40 pounds steam.

The standard sample shall be compounded according to the above formula and cured under the same conditions.

To be on-grade, the color of a freshly cut surface of the cured rubber compound containing the sample, shall be as white as a freshly cut surface of the compound containing the standard.

PHYSICAL TESTING METHOD NUMBER TWELVE
FOR THE PROPERTY OF:—TENSILE STRENGTH
AND ELONGATION.

Description:

The sample shall be incorporated in a rubber compound in the proportion of at least 10 volumes of pigment to 100 volumes of rubber.

Portions of the stock shall be mold-cured for varying times at a constant steam pressure and tested for tensile strength and elongation in the usual way.

The standard sample shall be compounded, cured and tested under identical conditions.

To be on-grade the tensile strength and elongation of the cured rubber compound containing the sample, in the region of the optimum cure, shall not deviate from that of the compound containing the standard, by an amount greater than 10 per cent plus or minus.

On account of the limitations in the methods, of testing compounded rubber, no sample shall be declared off grade without the confirmatory evidence of two out of three successive tests, starting in each case with a fresh portion of the pigment.

PHYSICAL TESTING METHOD NUMBER THIRTEEN
FOR THE PROPERTY OF:—ABRASION RESISTANCE.

Description:

Cured rubber stock containing the sample at the approximate optimum cure shall be selected in accordance with the method outlined in Testing Method No. 12.

Cured rubber stock containing the standard in a similar compound shall be selected in the same way.

The two stocks shall be tested against each other on a standard type of abrasion machine.

To be on-grade the abrasion resistance of the rubber compound containing the sample shall not deviate from that of the compound containing the standard by an amount greater than 10 per cent plus or minus.

PHYSICAL TESTING METHOD NUMBER FOURTEEN
FOR THE PROPERTY OF:—RATE OF CURE.

Description:

The criterion of the optimum cure shall be the maximum tensile product.

To be on-grade the time required to reach the optimum cure by the rubber compound containing the sample, shall not vary in minutes from that of the compound containing the standard, by more than 20 per cent plus or minus.

PHYSICAL TESTING METHOD NUMBER FIFTEEN
FOR THE PROPERTY OF:—PARTICLE SIZE.

Description:

A standard method of photomicrographic measurements of the individual particles shall be determined and the sample subjected to comparative measurements with the standard.

CHAPTER 10

STANDARD ANALYTICAL METHODS FOR TESTING THE CHEMICAL PROPERTIES OF ZINC OXIDE

ANALYTICAL TESTING METHOD NUMBER ONE FOR THE CONSTITUENT:—CHLORINE.

Description:

Ten grams of the sample are covered with water and 10 c.c. of N/10 AgNO_3 solution, which has been standardized against pure NaCl , added. Forty c.c. of concentrated HNO_3 are added and the solution boiled until nitrous fumes are removed. It is then cooled, 5 c.c. of ferric nitrate solution (1 : 6) added and the solution titrated to a faint pink with N/10 ammonium sulfocyanide (NH_4CNS). A blank shall be run with the same reagents, to determine the relative strengths of the solutions.

ANALYTICAL TESTING METHOD NUMBER Two FOR THE CONSTITUENT:— H_2O SOLUBLE SALTS.

Description:

Five grams of the sample are shaken in a 500 c.c. graduated flask for ten minutes with 250 c.c. of water at room temperature. The solution is made up to exactly 500 c.c. and filtered through dry paper. One hundred c.c. of the clear filtrate are measured out, poured into a weighed platinum dish, and evaporated to dryness on a sand-bath, the contents being protected from dust. The residue is dried for one or two hours at 110° C., cooled and weighed rapidly. The increase in weight represents the water soluble salts.

ANALYTICAL TESTING METHOD NUMBER THREE
FOR THE CONSTITUENT:—H₂O AT 110° C.

Description:

From 2 to 5 grams of the sample, which have been weighed in a glass-stoppered weighing bottle, are dried in an oven kept at practically constant temperature of 105° to 110° C. After cooling, the bottle is stoppered and weighed with its contents. The sample is returned to the oven for another hour and again cooled, stoppered and weighed as before. If there has been further loss of weight, this procedure is repeated until a constant weight is reached. The loss of weight in drying is the H₂O content at 110° C.

ANALYTICAL TESTING METHOD NUMBER FOUR
FOR THE CONSTITUENT:—INSOLUBLE IN HCl.

Description:

A sample of 10 grams is treated with 25 c.c. of water and 25 c.c. of hydrochloric acid and evaporated to dryness. The residue is taken up with 50 c.c. of 1 : 4 hydrochloric acid and the insoluble filtered off and thoroughly washed with 1 : 4 hydrochloric and then with boiling water. It is further washed with hot ammonium acetate solution and again with boiling water. The insoluble is then burned off and weighed.

ANALYTICAL TESTING METHOD NUMBER FIVE
FOR THE CONSTITUENT:—TOTAL SULPHUR AS SO₃.

Description:

A sample of 13.75 grams is treated with 5–10 c.c. of bromine water and then dissolved in 40 c.c. of concentrated HCl. The solution is boiled until the bromine has been expelled and then cooled. A small strip of aluminum is added and the solution heated gently to throw out the lead. The lead and insoluble matter are filtered off, the filtrate neutralized with ammonia, then made slightly acid with hydrochloric. The solution is heated to boiling and a slight excess of hot 10 per cent BaCl₂ solution added drop by drop with constant stirring. After standing in a warm place for two hours or longer, it is filtered through a previously ignited Gooch crucible. The BaSO₄ on the crucible is washed well with hot water, ignited in a muffle for thirty minutes, cooled and weighed, and the weight calculated to SO₃.

ANALYTICAL TESTING METHOD NUMBER SIX
FOR THE CONSTITUENTS:— PbSO_4 (Total PbO as).

Description:

A sample of 2 grams for 5 per cent leaded zinc oxide and $\frac{1}{2}$ gram for 35 per cent leaded zinc oxide shall be placed in 50 c.c. of water, 45 c.c. of concentrated HNO_3 added and the solution boiled. 50 c.c. more water and a few drops of dilute AgNO_3 shall be added. The solution shall be placed in a Frary rapid electrolytic stand, using a perforated platinum cylinder anode, and electrolyzed for forty-five minutes at $3\frac{1}{2}$ amperes. At the end of this time the anode shall be removed quickly, washed with water, dried at 180° C . for twenty minutes, cooled and desiccated. The deposit shall be weighed as PbO_2 and calculated to PbSO_4 . In the case of 35 per cent leaded zinc oxide a factor of 1.24 instead of 1.27 shall be used in the calculation.

ANALYTICAL TESTING METHOD NUMBER SEVEN
FOR THE CONSTITUENT:— PbSO_4 (Total PbO as).

Description:

A sample of 2 grams is treated (for 35 per cent leaded zinc oxide the sample is 1 gram) with 100 c.c. of water and 25 c.c. of concentrated HCl , and the insoluble matter filtered off. After the addition of 25 c.c. of 1 : 1 H_2SO_4 , the filtrate is evaporated until dense fumes come off, when 200 c.c. of water are added. The solution is heated until all soluble salts are dissolved, cooled, and 30 c.c. of 95 per cent alcohol added. It is allowed to stand overnight. The precipitate PbSO_4 is filtered off, washed first with 3 per cent H_2SO_4 and then with alcohol, dried and weighed.

ANALYTICAL TESTING METHOD NUMBER EIGHT
FOR THE CONSTITUENT:— PbSO_4 (Total PbO as).

Description:

A sample of 9.33 grams shall be placed in 50 c.c. of water, 45 c.c. of concentrated HNO_3 added and the solution boiled. 50 c.c. more water and a few drops of dilute AgNO_3 shall be added. The solution is placed on an electrolytic stand, using a solid sheet platinum anode, and electrolyzed for two hours at 1 ampere. At the end of this time the anode shall be removed quickly, washed with water, dried at 180° C . for twenty minutes, cooled and desiccated. The deposit shall be weighed as PbO_2 and calculated to PbSO_4 .

ANALYTICAL TESTING METHOD NUMBER NINE
FOR THE CONSTITUENT:—LEAD.

Description:

Thirty grams of the sample shall be weighed into a low 400 c.c. beaker, and 100 c.c. of water, and 80 c.c. of concentrated nitric added. After boiling for fifteen minutes the solution shall be diluted to about 350 c.c. Add a few drops of silver nitrate solution and electrolyze for at least five hours (overnight is preferable.)

Use a solid sheet cylindrical anode with a current density of 1 ampere when the deposit is desired in five hours. Use only $\frac{1}{2}$ ampere if deposition is to continue overnight.

ANALYTICAL TESTING METHOD NUMBER TEN
FOR THE CONSTITUENT:—ZINC OXIDE.

Description:

Digest 1.5 grams of freshly ignited zinc oxide, accurately weighed, with 50 c.c. of normal sulphuric acid until solution is complete. Then titrate the excess sulphuric acid with normal potassium hydroxide, using methyl orange as an indicator.

Each cubic centimeter of the above normal sulphuric acid used corresponds to 0.0407 gram of zinc oxide. Each gram of freshly ignited zinc oxide must correspond to not less than 24.3 c.c. of normal sulphuric acid.

ANALYTICAL TESTING METHOD NUMBER ELEVEN.
FOR THE CONSTITUENT:—H₂O SOLUBLE ALKALI.

Description:

Digest 1 gram of zinc oxide in a small flask with 10 c.c. of boiling distilled water. Add two drops of phenolphthalein indicator. Not more than 1 c.c. of tenth normal hydrochloric acid shall be required to discharge any red color that may be produced.

ANALYTICAL TESTING METHOD NUMBER TWELVE
FOR THE CONSTITUENT:—TOTAL HEAVY METALS.

Description:

Weigh 1 gram of zinc oxide into a test-tube of about 40 c.c. capacity and about 2.5 cms. in diameter, add about 10 c.c. of distilled water and dissolve with a very slight excess of hydrochloric acid. Heat the solution to about 50° C., add an equal volume of freshly prepared saturated solution of hydrogen sulphide in water. Stopper the test-tube, thoroughly mix the contents and allow to stand for half an hour in a moderately warm place (about 35° C.). At the end of this time the mixture should still possess the odor of hydrogen sulphide; if not, it shall be thoroughly saturated with the gas and again set aside for half an hour. The color produced shall not be greater than that observed by a blank test made in the same manner and with the same quantities of the reagents (omitting the zinc oxide). The solution shall be viewed crosswise by reflected light while held against white surfaces. A slight turbidity due to separation of sulphur from the hydrogen may occur.

ANALYTICAL TESTING METHOD NUMBER THIRTEEN
FOR THE CONSTITUENT:— SO_2 (Red. Power Equiv. to).*Description:*

A sample of 3.2 grams is treated with 100 c.c. of cold water and 100 c.c. of boiling water and some starch solution added as an indicator. Iodine solution (N/100) is run in until the end point is near, when 25 c.c. of concentrated hydrochloric acid are added and the titration with the iodine solution finished.

ANALYTICAL TESTING METHOD NUMBER FOURTEEN
FOR THE CONSTITUENT:—INSOLUBLE IN ACETIC ACID.*Description:*

Ten grams of zinc oxide shall be weighed into a 250 c.c. beaker; 100 c.c. of distilled water shall be added and the oxide stirred. 100 c.c. of 50 per cent acetic acid shall then be added, followed by 10 c.c. of strong ammonia water. The whole shall be stirred until all of the zinc oxide has dissolved, when it is allowed to stand for two or more hours. The insoluble residue is filtered on a weighed and dried Gooch crucible, and washed first with hot 10 per cent acetic acid, then with hot distilled water. The crucible is dried at 110° C. and reweighed; the gain in weight being the amount of insoluble matter in the 10 grams.

ANALYTICAL TESTING METHOD NUMBER FIFTEEN
FOR THE CONSTITUENT:—TOTAL SULPHUR AS SO₃.

Description:

A sample of 6.86 grams is mixed with 17 grams of sodium bicarbonate, 200 c.c. of water and 5 to 10 c.c. of bromine water added. The solution is boiled gently for fifteen minutes and set in a warm place until the precipitated carbonates settle. The latter are filtered off and washed thoroughly with hot water. The filtrate is boiled until the bromine is expelled and made slightly acid with hydrochloric. The solution is heated to boiling and a slight excess of hot 10 per cent BaCl₂ solution added drop by drop with constant stirring. After standing in a warm place for two hours or longer, it is filtered through a previously ignited Gooch crucible. The BaSO₄ on the crucible is washed well with hot water ignited in a muffle for thirty minutes, cooled and weighed, and the weight calculated to SO₃.

ANALYTICAL TESTING METHOD NUMBER SIXTEEN
FOR THE CONSTITUENT:—INSOLUBLE IN ACETIC ACID.

Description

Weigh 10 grams of the sample into a 250 c.c. beaker. Intimately mix with 50 c.c. of water, then add 50 c.c. of 50 per cent acetic acid. Allow to stand at room temperature for one-half hour, agitating frequently, then filter through a weighed Gooch crucible. Wash with luke-warm 10 per cent acetic acid, then with hot water. Dry at 110° C. and weigh.

ANALYTICAL TESTING METHOD NUMBER SEVENTEEN
FOR THE CONSTITUENT:—ARSENIC.

Description

Arsenic, reported as As_2O_3 , shall be determined by the Gutzeit method, using apparatus as shown in Scott's "Standard Method of Chemical Analysis," Second Edition, Revised, page 46.

Two grams of the sample shall be weighed into a 150 c.c. beaker and 50 c.c. of dilute sulphuric acid (1 volume arsenic free concentrated sulphuric acid, to 10 volumes of water) added and after complete solution has been effected, boiled for five minutes. Then 1 c.c. of ferrous sulphate solution (17.5 grams $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 100 c.c. water) and 1 c.c. of stannous chloride solution (40 per cent SnCl_2 in hydrochloric acid) shall be added.

The solution shall then be cooled, poured into the 60 c.c. bottle and the beaker washed with a minimum amount of water. The test strip of mercuric chloride paper shall then be placed in the upper tube, 2 grams of 20-mesh arsenic free zinc added to the solution in the bottle and the upper part of the apparatus immediately attached. After thirty minutes the depth of coloration of the test-paper shall be compared with standard test-papers from tests made using the same amounts of solutions and zinc but with known amounts of arsenic added. The mercuric chloride test-paper is made by moistening filter paper with 5 per cent solution of mercuric chloride, slightly pressing out the excess, drying, cutting accurately to size and keeping it tightly stoppered in dark glass bottles.

CHAPTER 11

UNITED STATES GOVERNMENT SPECIFICATIONS AND TEST- ING METHODS

UNITED STATES GOVERNMENT SPECIFICATION FOR ZINC OXIDE, DRY AND IN PASTE.

General:

Zinc oxide may be ordered in the form of dry pigment or paste ground in linseed oil. Purchases shall be made on the basis of net weight.

The pigment may be American Process zinc oxide, made direct from the ore, or French Process zinc oxide, made from spelter. The contract shall state which kind is desired.

The color and color strength when specified shall be equal to samples mutually agreed upon by buyer and seller.

The pigment shall meet the following specifications.

Coarse particles retained on Standard 325 screen.
Max. 1 per cent.

	American Process		French Process	
	Maximum Per cent	Minimum Per cent	Maximum Per cent	Minimum Per cent
Zinc oxide.....	98	99
Total sulphur.....	0.2	0.10	
Total impurities, moisture.	2.0	1.00	

The paste shall be made by thoroughly grinding the above pigment with pure raw or refined linseed oil. The paste shall not cake in the container and shall break readily in oil to form a smooth paint of brushing consistency.

The paste shall consist of:

	Maximum, Per Cent	Minimum, Per Cent
Pigment.....	86	82
Linseed oil.....	18	14
Coarse particles and skins.....	1.5	
Moisture and other volatile matter.....	0.5	

Sampling:

It is mutually agreed by the buyer and seller that a single package out of each lot of not more than 1000 packages shall be taken as representative of the whole.

With the dry pigment, this package is to be opened by the inspector and a sample of not less than 5 pounds taken at random from the contents and sent to the laboratory for test. When requested, a duplicate sample may be taken from the same package and delivered to the seller, and the inspector may take a third sample to hold for test in case of dispute.

With the paste, whenever possible, an original unopened container shall be sent to the laboratory; and when this is for any reason not done, the inspector shall determine by thoroughly testing with a paddle or spatula whether the material meets the requirement regarding not caking in the container. After assuring himself that the paste is not caked in the can, the inspector shall draw a sample of not less than

pounds of the thoroughly mixed paste, place it in a clean, dry metal or glass container which must be filled with the sample, closed with a tight cover, sealed, marked and sent to the laboratory for test with the inspector's report on caking in the container.

LABORATORY EXAMINATION OF DRY PIGMENT.

(1) *Color.*—Take 5 grams of the sample, add 1.5 c.c. of linseed oil, rub up on a stone slab or glass plate with a flat-bottomed glass or stone pestle or muller to a uniform smooth paste. Treat in a similar manner 5 grams of the standard sample of zinc oxide. Spread the two pastes side by side on a clear colorless glass plate and compare the colors. If the sample is as white as or whiter than the "standard," it passes this test. If the "standard" is whiter than the sample the material does not meet the specification.

(2) *Color Strength.*—Weigh accurately 0.01 gram of lampblack, place on a large glass plate or stone slab, add 0.2 c.c. of linseed oil, and rub with a flat-bottomed glass pestle or muller; then add exactly 10 grams of the sample and 2.5 c.c. of linseed oil, and grind with a circular motion of the muller 50 times; gather up with a sharp-edged spatula and grind twice more in a like manner, giving the pestle a uniform pressure. Treat another 0.01 gram of the same lampblack in the same manner except that 10 grams of the standard sample zinc oxide shall be used instead of the 10 grams of the sample. Spread the two pastes side by side on a glass microscope slide and compare the colors. If the sample is as light as or lighter in color than the "standard," it passes this test. If the "standard" is lighter in

color than the sample, the material does not meet the specification.

(3) *Coarse Particles*.—Dry in an oven at 105 to 110° C. a 325 mesh screen, cool and weigh accurately. Weigh 10 grams of the sample; dry at 100° C., transfer to a mortar, add 100 c.c. kerosene, thoroughly mix by gentle pressure with a pestle to break up the lumps, wash with kerosene through the screen, breaking up all of the lumps but not grinding. After washing with kerosene until all but the particles which are too coarse to pass through the screen have been washed through, wash all kerosene from the screen with either or petroleum ether, heat the screen for one hour at 105 to 110° C., cool and weigh.

(4) *Qualitative Analysis*.—Test for matter insoluble in hydrochloric acid, for lead, calcium, etc., by regular methods of qualitative analysis.

(5) *Zinc Oxide*.—With samples free from impurities ignite a weighed sample and calculate the residue as zinc oxide. With samples containing impurities, proceed as follows: Weigh accurately about 0.25 gram, transfer to a 400 c.c. beaker, moisten with alcohol, dissolve in 10 c.c. of hydrochloric acid and 20 c.c. of water and titrate with standard potassium ferrocyanide following the procedure used in standardizing this reagent.

(6) *Total Sulphur*.—Weigh accurately about 10 grams of the sample. Moisten with a few drops of alcohol, add 5 c.c. of bromine water (saturated solution of bromine), then concentrated hydrochloric acid in excess, boil to expel the bromine, and dilute to about 100 c.c. (Material complying with the specifications should all go into solution; if insoluble matter remains,

filter and examine by appropriate methods.) Make alkaline with ammonia, then just acid with hydrochloric acid, heat to boiling-point and add about 10 c.c. of hot barium chloride solution. Let stand several hours (overnight), filter on a weighed Gooch crucible, wash thoroughly with hot water dry, ignite, and weigh with BaSO_4 . Calculate S ($\text{BaS}\text{XO}_4 \cdot 0.1373 = \text{S}$).

LABORATORY EXAMINATION OF PASTE.

(1) *Caking in Container*.—When an original package is received in the laboratory, it shall be weighed, opened and stirred with a stiff spatula or paddle. The paste shall be no more difficult to break up and show no more caking than a normal good grade of zinc oxide paste. The paste shall be finally thoroughly mixed, removed from the container, the container wiped clean, and weighed. This weight subtracted from the weight of the original package gives the net weight of the contents. A portion of the thoroughly mixed paste shall be placed in a clean container and the portions for the remaining tests promptly weighed out.

(2) *Mixing with Linseed Oil*.—One hundred grams of the paste shall be placed in a cup, 35 c.c. of linseed oil added slowly with a careful stirring and mixing with a spatula or paddle. The resulting mixture must be smooth and of good brushing consistency.

(3) *Moisture and Other Volatile Matter*.—Weigh accurately from 3 to 5 grams of the paste into a tared flat-bottomed dish, about 5 cm. in diameter, spreading the paste over the bottom. Heat at 110° C . for one hour, cool and weigh. Calculate loss in weight as percentage of moisture and other volatile matter.

(4) *Per Cent Pigment.*—Weigh accurately about 15 grams of the paste into a weighed centrifuge tube. Add 20 to 30 c.c. of "extraction mixture" (see Reagents), mix thoroughly with a glass rod, wash the rod with more of the extraction mixture, and add sufficient of the reagent to make a total of 60 c.c. in the tube. Place the tube in the container of the centrifuge, surrounded with water, and counterbalance the container of the opposite arm with a similar tube or a tube of water. Whirl at a moderate speed until clear. Decant the supernatant liquid. Repeat the extraction twice with 40 c.c. portions of extraction mixture, and once with 40 c.c. of ether. After drawing off the ether, set the tube in a beaker of water at 80° C. or on top of a warm oven for ten minutes, then in an oven at 110° C. to 115° C. for two hours. Cool, weigh, and calculate percentage of pigment.

(5) *Examination of Pigment.*—Grind the pigment from (4) to a fine powder, pass through a No. 80 mesh screen to remove any "skins," preserve in a stoppered tube and apply tests Nos. 4, 5, and 6, of Laboratory Examination of Dry Pigment. If required, apply tests Nos. 1 and 2 also, with a portion of pigment extracted from the standard paste in exactly the same manner as in extracting the sample.

(6) *Preparation of Fatty Acids.*—To about 25 grams of the paste in a porcelain casserole add 15 c.c. aqueous sodium hydroxide (see Reagents), add 75 c.c. of ethyl alcohol, mix and heat uncovered on a steam bath until saponification is complete (about one hour). Add 100 c.c. of water, boil, add an excess of sulphuric acid of specific gravity 1.2 (8 to 10 c.c. will usually suffice), boil, stir, and transfer to a separatory funnel to which

some water has been previously added. Draw off as much as possible of the acid aqueous layer, wash once with water; then add 50 c.c. of water and 50 c.c. of ether. Shake very gently with a whirling motion to dissolve the fatty acids in the ether, but not violently, so as to avoid forming an emulsion. Draw off the aqueous layer and wash the ether layer with one 15 c.c. portion of water and then with 5 c.c. portions of water until free from sulphuric acid. Then draw off completely the water layer. Transfer the ether solution to a dry flask, and add 25 to 50 grams of anhydrous sodium sulphate. Stopper the flask and let stand with occasional shaking at a temperature below 25° C. until the water is completely removed from the ether solution, which will be shown by the solution becoming perfectly clear above the solid sodium sulphate. Decant this clear solution (if necessary through a dry filter paper) into a dry 100 c.c. Erlenmeyer flask. Pass a rapid current of dry air (pass through CaCl_2 tower) into the mouth of the Erlenmeyer flask and heat to a temperature below 75° C. on a dry hot plate until the ether is entirely driven off. The fatty acids prepared as above should be kept in a stoppered flask and examined at once. *Note.*—It is important to follow all of the details, since ether generally contains alcohol and after washing with water always contains water. It is very difficult to remove water and alcohol by evaporation from fatty acids, but the washing of the ether solution and subsequent drying with anhydrous sodium sulphate removed both water and alcohol. Ether, in the absence of water and alcohol, is easily removed from the fatty acids by gentle heat.

(7) *Tests for Mineral Oil and Other Unsaponifiable*

Matter.—Place 10 drops of the fatty acid (6) in a 50 c.c. test-tube, add 5 c.c. of alcoholic soda (see Reagents), boil vigorously for five minutes, add 40 c.c. of water, and mix; a clear solution indicates that not more than traces of unsaponifiable matter are present. If the solution is not clear, the oil is not pure linseed oil.

(8) *Iodine Number of Fatty Acids.*—Place a small quantity of the fatty acids (6) in a small weighing burette or beaker. Weigh accurately. Transfer by dropping about 0.15 gram (0.10 to 0.20 gram) to a 500 c.c. bottle having well-ground glass stopper, or an Erlenmeyer flask having a specially flanged neck for the iodine test. Reweigh the burette or beaker and determine the amount of the sample used. Add 10 c.c. of chloroform. Whirl the bottle to dissolve the sample. Add 10 c.c. of chloroform to two empty bottles like that used for the sample. Add to each bottle 25 c.c. of the Hanus solution (see Reagents) and let stand, with occasional shaking, for one-half hour. Add 10 c.c. of the 15 per cent potassium-iodide solution and 100 c.c. water, and titrate with standard sodium thiosulphate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 c.c. From the difference between the average of the blank titrations and the titration on the sample and the iodine value of the thiosulphate solution, calculate the iodine number of the sample tested. (Iodine number in centigrams of iodine to 1 gram of sample.) If the iodine number is less than 170, the oil does not meet the specification.

(9) *Coarse Particles and "Skins."*—Weigh an amount of the paste containing 10 grams of pigment (see above), add kerosene, and wash through a No. 325 screen as in

Dry Pigment Examination No. 3. The residue is reported as "coarse particles and skins."

REAGENTS.

(1) *Extraction Mixture*.—

10 volumes of ether (ethyl ether)
6 volumes of benzol
4 volumes methyl alcohol
1 volume of acetone.

(2) *Aqueous Sodium Hydroxide*.—Dissolve 100 grams sodium hydroxide in distilled water and dilute to 300 c.c.

(3) *Standard Sodium Thiosulphate Solution*.—Dissolve pure sodium thiosulphate in distilled water that has been well boiled to free it from carbon dioxide, in the proportion of 24.83 grams crystallized sodium thiosulphate to 1000 c.c. of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Analytical Chemistry, Treadwell-Hall, Vol. 2, 3d edition, page 646.) This solution will be approximately decinormal, and it is best to leave it as it is after determining the exact iodine value rather than to attempt to adjust it to exactly decinormal. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(4) *Starch Solution*.—Stir up 2 or 3 grams of potato starch or 5 grams soluble starch with 100 c.c. of 1 per cent salicylic acid solution, add 300 c.c. to 400 c.c. boiling water, and boil the mixture until the starch is practically dissolved, then dilute to 1 liter.

(5) *Potassium Iodide Solution.*—Dissolve 150 grams of potassium iodide free from iodate in distilled water and dilute to 1000 c.c.

(6) *Hanus Solution.*—Dissolve 13.2 grams of iodine in 1000 c.c. of 99.5 per cent glacial acetic acid which will not reduce chromic acid. Add enough bromine to double the halogen content, determine by titration (3 c.c. bromine is about the proper amount). The iodine may be dissolved by the aid of heat, but the solution should be cold when the bromine is added.

(7) *Alcoholic Sodium Hydroxide Solution.*—Dissolve pure sodium hydroxide in 95 per cent ethyl alcohol in the proportion of about 22 grams per 1000 c.c. Let stand in a stoppered bottle. Decant the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used, and it will keep colorless longer if the alcohol is previously treated with sodium hydroxide (about 80 grams to 1000 c.c.) kept at about 50° C. for fifteen days and then distilled.

(8) *Uranyl Indicator for Zinc Titration.*—A 5 per cent solution of uranyl nitrate in water or a 5 per cent solution of uranyl acetate in water made slightly acid with acetic acid.

(9) *Standard Potassium Ferrocyanide.*—Dissolve 22 grams of the pure salt in water and dilute to 1000 c.c. To standardize transfer about 0.2 gram (accurately weighed) of pure metallic zinc or freshly ignited pure zinc oxide to a 400 c.c. beaker. Dissolve in 10 c.c. hydrochloric acid and 20 c.c. water. Drop in a small piece of litmus paper, add ammonium hydroxide until slightly alkaline, then add hydrochloric acid until just acid, and then add 3 c.c. strong hydrochloric acid.

Dilute to about 250 c.c. with hot water and heat nearly to boiling. Run in the ferrocyanide solution slowly from a burette with constant stirring until a drop tested on a white porcelain plate with a drop of uranyl indicator shows a brown tinge after standing a minute. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of ferrocyanide solution required for the blank should be subtracted from the amounts used in the standardization and in titration of the sample. The standardization must be made under the same conditions of temperature, volume, and acidity as obtained when the sample is titrated.

(10) *Barium Chloride Solution*.—Dissolve 100 grams of pure crystallized barium chloride in water and dilute to 1000 c.c.

UNITED STATES GOVERNMENT SPECIFICATION
FOR ZINC OXIDE (LEADED ZINC OXIDE)
DRY OR IN PASTE.

General.

Leaded zinc oxide, frequently known as leaded zinc, consists of zinc oxide and varying amounts of lead compounds. It may be ordered in the form of dry pigment or paste ground in linseed oil. Purchases shall be made on the basis of net weight.

The pigment may be high-leaded zinc oxide or low-leaded zinc oxide. The contract shall state what kind is desired. The color and color strength when specified shall be equal to samples mutually agreed upon by the buyer and the seller.

The pigment shall meet the following requirements:

	High Leaded		Low Leaded	
	Max. Per Cent	Min. Per Cent	Max. Per Cent	Min. Per Cent
Zinc oxide (ZnO).....	60	93
Water soluble salts.....	1.0	1.0	
Total impurities, inc. moisture.....	1.5	1.5	
The balance to be normal or basic lead sulphate.....				

Coarse particles retained on No. 325 mesh screen.
Max. 1.0 per cent min.

The paste shall be made by thoroughly grinding the pigment with pure raw or refined linseed oil. The paste shall not cake in the container and shall break up readily in oil to form a smooth paint of brushing consistency. The paste shall consist of:

	Maximum, Per Cent	Minimum, Per Cent
Pigment.....	88.0	
Linseed oil.....	12.0
Moisture and other volatile matter.....	.5	
Coarse particles and "skins".....	1.5	

SAMPLING.

The sampling method and procedure for leaded zinc oxide is the same as given for lead free zinc oxide.

LABORATORY EXAMINATION OF DRY PIGMENT

(1) *Color*.—Use the same test as outlined in laboratory procedure for lead free zinc oxide.

(2) *Color Strength*.—Use the same test as outlined in laboratory procedure for lead free zinc oxide.

(3) *Qualitative Analysis*.—Test for matter insoluble in hydrochloric acid, lead, calcium, carbon dioxide, etc., by regular methods of qualitative analysis.

(4) *Moisture*.—Place 1 gram of the sample in a wide-mouthed short weighing tube provided with a glass stopper. Heat with stopper removed for two hours at a temperature between 100 to 105° C. Insert stopper, cool and weigh. Calculate loss in weight as moisture.

(5) *Water Soluble Salts*.—To 10 grams of the pigment in a 500 c.c. volumetric flask add 200 c.c. of water, boil for five minutes, nearly fill the flask with hot water, allow to cool, fill to mark, mix, filter through a dry paper, discard the first 50 c.c. of filtrate, transfer 100 c.c. of the filtrate (corresponding to 2 grams of the sample) to a weighing dish, evaporate to dryness, heat for one hour in an oven at 105 to 110° C., cool, and weigh, calculate the percentage of water soluble salts.

(6) *Zinc Oxide*.—Weigh accurately about 0.3 gram of the pigment, transfer to a 400 c.c. beaker, add 30 c.c. of hydrochloric acid (1 : 2), boil for two or three minutes, add 200 c.c. of water and a small piece of litmus paper; add ammonium hydroxide until slightly alkaline,

render just acid with hydrochloric acid, then add 3 c.c. of strong hydrochloric acid, heat nearly to boiling, and titrate with standard potassium ferrocyanide as in the standardizing solution. (See Reagent No. 4.) Calculate total zinc as ZnO.

■ (7) *Calculations*.—If, as will be the case with material complying with the specification, no metal but zinc and lead are found by qualitative analysis tests, add the percentage of ZnO, moisture, and water soluble salts and subtract the sum from 100. Call the remainder "normal and basic lead sulphate."

LABORATORY EXAMINATION OF PASTE

(1) *Caking in the Container*.—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(2) *Mixing with Linseed Oil*.—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(3) *Moisture and Other Volatile Matter*.—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(4) *Percentage of Pigment*.—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(5) *Examination of Pigment*.—Grind the pigment from (4) to a fine powder, pass through a No. 80 mesh screen to remove any "skins," preserve in a stoppered tube, and apply tests Nos. 4, 6, 7 and 8 of the procedure outlined in Laboratory Examination of Dry Pigment. If required, apply tests 1 and 2 of that procedure in comparison with a portion of pigment extracted from

the standard paste in exactly the same manner as in extracting the sample.

(6) *Preparation of Fatty Acids.*—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(7) *Test for Mineral Oil and other Unsaponifiable Matter.*—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(8) *Iodine Number of Fatty Acids.*—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

(9) *Coarse Particles or "Skins."*—Use the same test as outlined in laboratory procedure for lead-free zinc oxide in paste.

REAGENTS

(1) *Uranyl Indicator for Zinc Titration.*—A 5 per cent solution of uranyl nitrate in water or a 5 per cent solution of uranyl acetate in water made slightly acid with acetic acid.

(2) *Standard Potassium Ferrocyanide.*—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

(3) *Barium Chloride Solution.*—Dissolve 100 grams of pure crystallized barium chloride in water and dilute to 1000 c.c.

(4) *Standard Sodium Thiosulphate Solution.*—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

(5) *Starch Solution.*—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

(6) *Extraction Mixture*.—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

(7) *Aqueous Sodium Hydroxide*.—Dissolve 100 grams of sodium hydroxide in distilled water and dilute to 300 c.c.

(8) *Potassium Iodide Solution*.—Dissolve 150 grams of potassium iodide free from iodate, in distilled water and dilute to 1000 c.c.

(9) *Hanus Solution*.—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

(10) *Alcoholic Sodium Hydroxide Solution*.—Use the same procedure as outlined under Reagents for lead-free zinc oxide.

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